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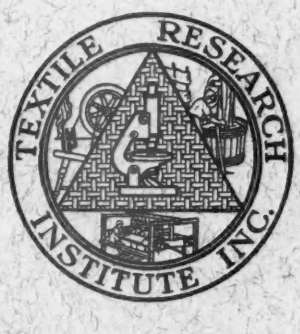

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TEXTILE RESEARCH JOURNAL

VOL. XVII, No. 5 May, 1947

Stress Relaxation of Cotton and Rayon Cords at Constant Length*

Edward G. Burleigh and Helmut Wakeham**

Southern Regional Research Laboratory,† New Orleans, Louisiana

RECENT advances in the science of high polymers have resulted in the development of theories which permit interpretation of textile properties in terms of molecular quantities. These theories make possible evaluation of some of the molecular constants required to correlate satisfactorily the physical and chemical behavior of a textile fiber with the properties of the molecules of which it is composed. The present paper demonstrates the application of stress-relaxation data to the Tobolsky-Eyring [26] theory of mechanical properties of high-polymeric materials for the purpose of determining certain molecular constants characteristic of cellulose fibers.

The investigation of stress relaxation at constant deformation of textile materials has been seriously neglected despite the advantages of such a study in adding to our knowledge of textile behavior. Experimental data which have been reported for glass filaments [9], rubber [2, 15, 21, 25, 27], gelatin gels [4], lead [28], steel [29], and polyvinyl acetate [17] have only limited application to the problem of textiles. Smith and Eisenschitz [24] investigated stress relaxations of viscose rayon, cellulose acetate, and silk. They attempted to interpret their results in terms of the Boltzmann "after-effect theory," which they found permits only an "approximation to the actual behavior of rayon." On the other hand, the reviews by Press [23], Leaderman [10], and Lyons [11] indicate a wealth of flow and creep-recovery data which, because of the complexity of the phenomena involved, cannot be used in evaluating molecular constants as readily as stress-relaxation data. As Lyons points out, stress relaxation eliminates strain as a variable and permits investigation of "the adjustments taking place at the micellar and molecular levels, without the complication of concomitant deformation in the fiber substance."

The present report is of just such an investigation. In it a new method for evaluating stress relaxation is described, and the results obtained are interpreted in terms of current molecular concepts of the structure of cellulose fibers.

Theory

Three types of motion in a fiber are postulated in the reaction-rate theory of elasto-viscous behavior as outlined by Tobolsky and Eyring [25, 26]: motion due to decay of the primary structural elements of the fiber network; motion due to relaxation of secondary cross bonds permitting units of the network structure to slip on the application of stress; and motion of the mobile segments of the long molecules or groups of molecules. Equations based on a modi-

* Read before the Fiber Society, New Orleans, February 20, 1947.

** Dr. Wakeham's present address: Institute of Textile Technology, Charlottesville, Virginia.

† One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

fication of the Maxwell-Wiechert theory have been written for each of these types of motion. Finally, the theory has been extended and applied to textile fibers as outlined by Eyring and coworkers [6] at the Textile Foundation Laboratories.

In the experimental determination of stress relaxation, the substance is rapidly stretched to a given length which is held fixed while the stress decay with time is measured. In this type of experiment it is believed that the stress due to motion of the mobile segments decays very rapidly so that it is not observable. The stress due to the primary structural elements of the network units, on the other hand, decays very slowly, if at all. The observed stress decay is, then, due to the relaxation of secondary network bonds; and a study of stress relaxation under these conditions should yield information concerning the nature of such bonds.

The equation of motion for secondary bond relaxation is, in the notation of Tobolsky and Eyring [26],

$$\frac{ds}{dt} = \frac{1}{G}\frac{df}{dt} + A \sinh Bf, \tag{1}$$

where (ds/dt) is the rate of change of length with time, (df/dt) is the rate of change of force, f, with time, and G, A, and B are molecular constants defined as:

G = an elastic modulus of the substance,

$$A = 2n\lambda \frac{kT}{h} \exp(-\Delta F^{\ddagger}/RT), \tag{2}$$

and

$$B = \frac{\lambda}{2NkT}. \tag{3}$$

In this theory it is assumed that there are N parallel flowing units in a square centimeter of fiber cross section, that the distance between successive equilibrium positions of each flowing unit in the direction of stress is λ, that n is the average number of secondary bonds per centimeter which are involved in the movement of each flow unit, and that $\Delta F^{\ddagger}$ is the free energy of activation for movement from one equilibrium position to the next. The other terms in equations (2) and (3) are Boltzmann's constant, $k = 1.371 \times 10^{-16}$ erg/°C; Planck's constant, $h = 6.55 \times 10^{-27}$ erg sec.; the gas constant, $R = 1.987$ cal./mole; and the absolute temperature, T. The force f/N is then the average force on each flowing unit which tends to favor motion from one equilibrium position to another in the direction of stress.

Since in the stress-relaxation experiment with constant strain $(ds/dt) = 0$, equation (1) may be integrated to obtain

$$\tanh\left(\frac{Bf}{2}\right) = \tanh\left(\frac{Bf_0}{2}\right) e^{-k't}, \tag{4}$$

where f_0 is the initial stress and $k' = ABG$. Figure 1 is a graph of this hyperbolic tangent decay function where f/f_0 is plotted against the logarithm of $k't$ for different values of $Bf_0/2$.

The constants B and k' in equation (4) may be evaluated from the experimental relaxation curves of f/f_0 plotted against log t. Values of G are obtained from the experimentally determined elastic modulus of the material during a rapid, reversible stress-strain cycle. With these constants it then becomes possible to calculate the number of flow units, N, per square centimeter of fiber cross section and $\Delta F^{\ddagger}$, providing values may be assigned to the displacement distance, λ, and to the number of bonds, n, per centimeter length of flow unit. These values are obtained from a consideration of cellulose fiber structure and molecular coordination.

Present concepts of the molecular architecture of the cellulose fiber may be summarized as follows [7, 18, 22]: The fiber consists of a structure of long cellulose chains which lie roughly parallel to the long axis of the fiber. In localized regions of the fiber the chains are orientated with respect to each other in such a way as to form a crystalline lattice. The fraction of cellulose which is thus associated in crystalline regions varies from fiber to fiber. For native cotton cellulose this fraction is estimated to be between 60 and 95 percent and for regenerated cellulose between 25 and 85 percent [1, 5, 7, 13, 20]. Because the chain length is but a fraction of the fiber length, stress from one end of the fiber to the other must be transmitted from one chain to its neighbors by means of intermolecular bonds or forces. These forces are probably strongest in the crystalline regions where the chains lie in closest proximity to each other. As stress is applied to the fiber and as slack in the noncrystalline portions is taken up, forces are exerted to cause slippage of the cellulose chains with respect to each other. This slippage is believed to be responsible for stress relaxation under conditions of constant deformation of the fiber.

The dimension of the unit cell of crystalline cellulose in the direction parallel to the cellulose chain is the length of the cellobiose unit, the repeating element in the polymer chain [16]. The cell structure and molecular coordination between chains are such that if one chain moves in the direction of its long axis with respect to its neighbors, it will not reach a position of equal stability until it has moved the length of the cellobiose unit—that is, 10.3 Å. This should, then, be the minimum distance between equilibrium positions of the chain if the crystalline structure is to be preserved. Hence, we arrive at $\lambda = 10.3 \times 10^{-8}$ cm.

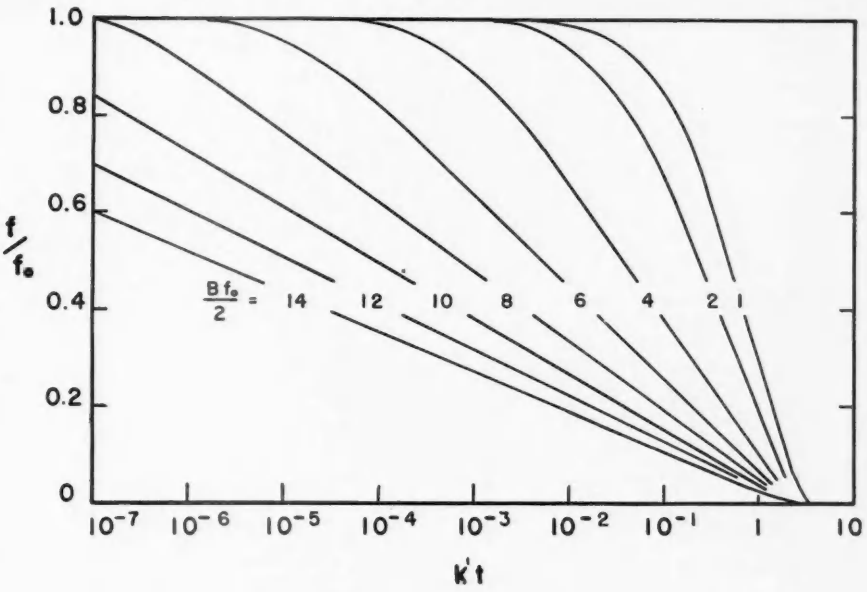

FIG. 1. *Hyperbolic tangent decay curves for values of* $Bf_0/2$.

The number of bonds per centimeter, n, may be evaluated from Peirce's investigation [22] of the molecular coordination in both native and regenerated cellulose. He assigns hydroxyl bonds to positions between the chains in the 002 planes of native cellulose and in the $10\bar{1}$ planes of the regenerated cellulose. These hydroxyl bonds are the secondary bonds largely responsible for restricting movement of the flow units. In native cellulose there are postulated 4 such bonds per cellobiose unit for those chains in the centers of the unit cells and 6 bonds per cellobiose unit for the chains at the corners of the cells. The average value will then be 5 bonds per cellobiose unit because there are an equal number of units in each position of the cell. Hence, for native cellulose, $n = 5/10.3 \times 10^{-8}$. From similar considerations for the regenerated cellulose, $n = 10/10.3 \times 10^{-8}$.

The probability that the flow unit consists of several chains held together by hydroxyl bonds and van der Waals forces, instead of single chains as assumed in the foregoing evaluations of λ and n, does not seriously affect the application of the Tobolsky-Eyring theory. The distance between equilibrium positions of multiple-chain flow units would still be the length of the cellobiose unit. Although the combination of chains making up the flow unit might result in larger values of n, the minimum values for n would still be those indicated. Since stress slippage may be expected to occur along lines of least resistance—that is, where the secondary cross links are at a minimum—situations in which n is a minimum will probably predominate. Furthermore, when f_0 is large and when the fiber structure is swollen with water so that the interchain hydroxyl bonds are weakened, it seems possible that N may approach the actual number of chains present. Under such conditions the chains may be expected to flow as individual units.

Experimental Data

The bonded metalectric strain gage is ideally suited for investigations in which the problem is one of either continuous or intermittent measurement of stress [8] over a period of time. Such is the problem in this investigation, in which rapidly decaying stress in a strained cord must be measured as a function of time with the added condition imposed that the length must be held constant. This condition was met by using as the measuring element a strain gage on a metal weighbar attached to the cord; since the elongation in the weighbar was extremely small in comparison with that in the cord, the process of relaxation in the cord can be considered one of essentially constant length.

The gage used with most success in this work was the Baldwin SR-4 strain gage, type A-11, which has a resistance of 120 ohms and a gage factor of 2.10. The gage factor was high enough to give

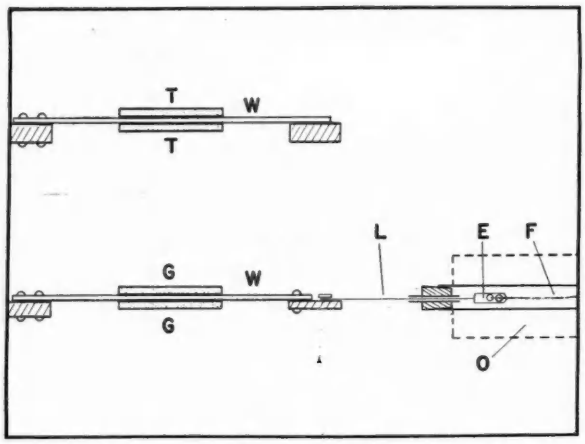

FIG. 2. *Schematic diagram of relaxation apparatus. G—active gage, T—temperature compensator gage, W—brass weighbar, L—steel-wire leader, E—brass tie, F—cord, and O—oven.*

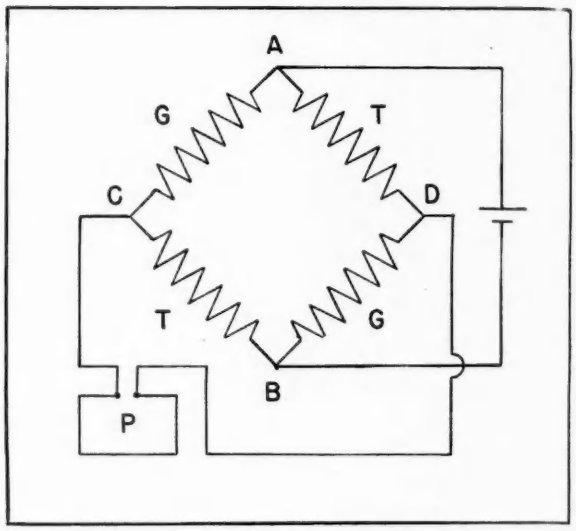

FIG. 3. *Diagram of strain-gage circuit. G—active gage, T—temperature compensator gage, P—potentiometer, A–B—points across which input e.m.f. is measured, and C–D—points across which output e.m.f. is measured.*

good sensitivity when mounted on a thin weighbar, and the temperature coefficient was sufficiently low to obviate the necessity of precise thermal control beyond that obtainable with an ordinary air-conditioning unit. Room temperature was actually controlled within $\pm 2°F$.

The gages were mounted longitudinally on weighbars cut from 0.003-inch brass shimstock $4\frac{1}{2}$ inches $\times \frac{3}{8}$ inch, a gage cemented to each side of the strip. Two of these strips, or 4 gages, were assembled to make up a gage network, or measuring unit. One strip was placed horizontally in the apparatus (Figure 2) to carry the tension; hence, the 2 gages bonded to it were "active" gages. Another strip placed directly above and parallel to the first carried no load, and the 2 gages bonded to it acted as temperature compensators. The 4 gages of the measuring unit were wired so as to make up the 4 arms of a Wheatstone bridge (Figure 3), the unbalance of which was a measure of the resistance changes in the active gages brought about when tension was applied to the active strip. The cord to be tested was attached to the free end of the weighbar by means of a steel wire leader, *L* (Figure 2), with stirrup-like brass tie, and the desired tension was applied to the other end of the cord through a similar tie by weights suspended over a ball-bearing pulley. The cord and wire leaders were aligned with the longitudinal axis of the weighbar so that all the load applied to the end of the cord was conveyed to the active gages. The cord itself was inside a long tube in an insulated chamber or oven in which the temperature and humidity conditions could be controlled.

The apparatus consisted of a battery of 6 such measuring units so that 6 cords could be tested simultaneously (Figure 4).

A constant electromotive force was applied to the Wheatstone bridge at points A and B (Figure 3), and the potentiometer measuring circuit measured the output of the gages across points C and D. Through a double-pole, double-throw switch the potentiometer circuit could also be utilized to measure the applied e.m.f. across points A and B.

By calibration of the gages under static loads it was confirmed that the e.m.f. output was essentially linear with respect to load [3]. The applied e.m.f. was adjusted through a variable resistance so that the sensitivity of the gage was very nearly 2 pounds per millivolt. The applied e.m.f. required to achieve this sensitivity was in each case approximately 10.5 volts.

It was found that the performance of the gages was materially improved by a "conditioning" for several hours under the load at which the cords were to be tested.

Since the relaxation of stress in a cord is initially a very rapid process, measurements of the gage output during the first few seconds of the relaxation had to be made with considerably greater speed than

is possible with a manually operated potentiometer. Therefore, the early stages of the relaxation were followed by the deflection of the null-indicating galvanometer of the potentiometer circuit, the sensitivity of which was properly adjusted by appropriate series and parallel resistances. By simultaneously photographing with a movie camera the galvanometer and an electric timer having a large sweep second hand, a continuous record was made of the galvanometer deflection throughout the first few seconds of the relaxation. Thus, at any elapsed time the galvanometer deflection could be read from the film record, and from that deflection the drop in e.m.f. output of the gage could be determined. After the first 60 seconds, the relaxation was proceeding slowly enough to be followed by direct readings on the potentiometer.

The cord samples to be tested, 50 centimeters in length, were preconditioned before being inserted in the apparatus. For the dry experiments they were first dried in a vacuum at room temperature for several hours and then placed in the tubes of the test oven under a stream of dry air. Higher moisture contents were attained by conditioning the cords at room humidity. Conditioning to 100 percent relative humidity was achieved by blowing moisture-saturated air through the oven tubes. After suitable conditioning the cords were rapidly stretched at 2 seconds before "zero" time and clamped in position at "zero" time. At the instant of clamping the galvanometer began its rapid deflection corresponding to stress decay in the cord. The record of time and gage e.m.f. was made as described earlier.

Relaxation experiments were made with two tire cords: one a wet-stretched cotton tire cord of 17/4/3 construction, a dry Grex number of 4446, and an oven-dry breaking strength of 2.24 g./Grex; the other a viscose rayon cord of 2200/2 construction, a dry Grex number of 5366, and an oven-dry breaking strength of 2.90 g./Grex. The vacuum-dried cords were relaxed at 25, 110, and 160°C with initial loads of 0.5 g./Grex, and at 25°C with initial loads of 1.0 g./Grex. The moist cords were all relaxed at 25°C with initial loads of 0.5 g./Grex. Several experimental relaxation runs, in some cases as many as nine, were made for each cord at each condition tested. Unfortunately it was not experimentally practical to extend the observations to higher or lower initial loads than 1.0 and 0.5 g./Grex, respectively. At appreciably higher loads too large a percentage of the cords broke upon loading; at lower loads the low sensitivity of the electric gages made the experimental error in stress determination proportionately large.

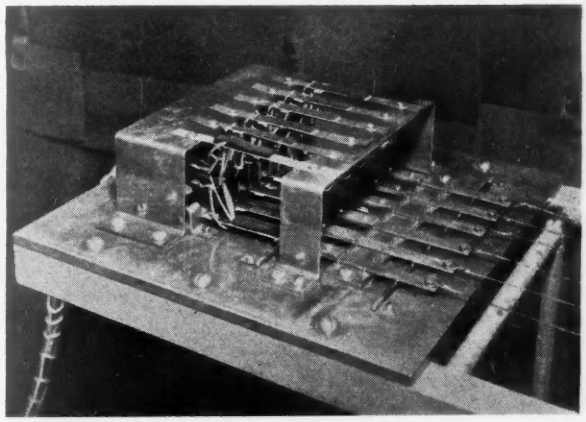

FIG. 4. *Photograph of strain-gage units used in stress-relaxation experiments.*

Results and Discussion

Typical relaxation curves at various conditions are shown in Figure 5 (cotton cord) and Figure 6 (rayon cord).

From the results of such relaxation experiments the constants shown in Table I were calculated. The initial force, f_0, and the number of cellulose chains, N_C, both per square centimeter of cross section, were evaluated from the Grex number of the cord and the density of crystalline cellulose (1.582 for cotton and 1.595 for rayon) [12, 18]. For a given experiment, the value of $Bf_0/2$ was found by comparing the slope of the experimental curve with the theoretical curves shown in Figure 1; similarly, k' was obtained from the measured displacement necessary to shift the experimental curve to its corresponding theoretical curve. The elastic modulus, G, at each condition for the cord was obtained from the observed modulus of the cord [30] corrected for the intra- and interfiber spaces by multiplying the observed modulus by the ratio of the density of crystalline cellulose to the apparent density of the cord calculated from the cord gage and Grex number. From these data and the values of n and λ described earlier, the constants A, B, N, and $\Delta F^{\ddagger}$ were calculated by means of equations (2), (3), and (4). In addition to these constants, Table I shows values of the ratio N_C/N, which is the average

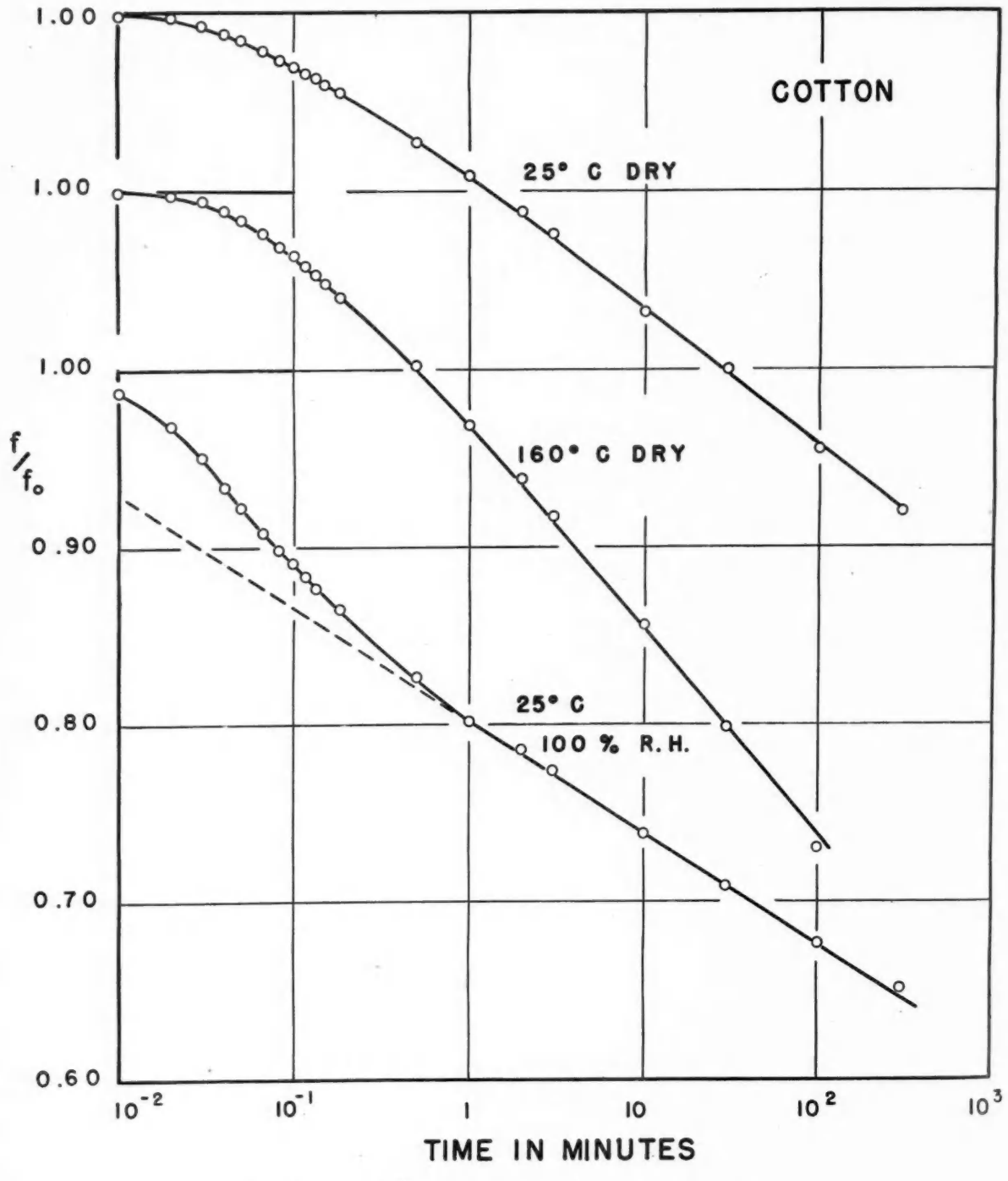

FIG. 5. *Experimental stress-relaxation curves for cotton.*

number of cellulose chains per flow unit for the cord during the relaxation process.

The authors in making these calculations have assumed that the observed stress relaxation is an intrafiber process and that the interfiber movement, or slippage, which takes place during the loading to maximum stress, ceases at the instant the cord is clamped at constant length. It seems unlikely that

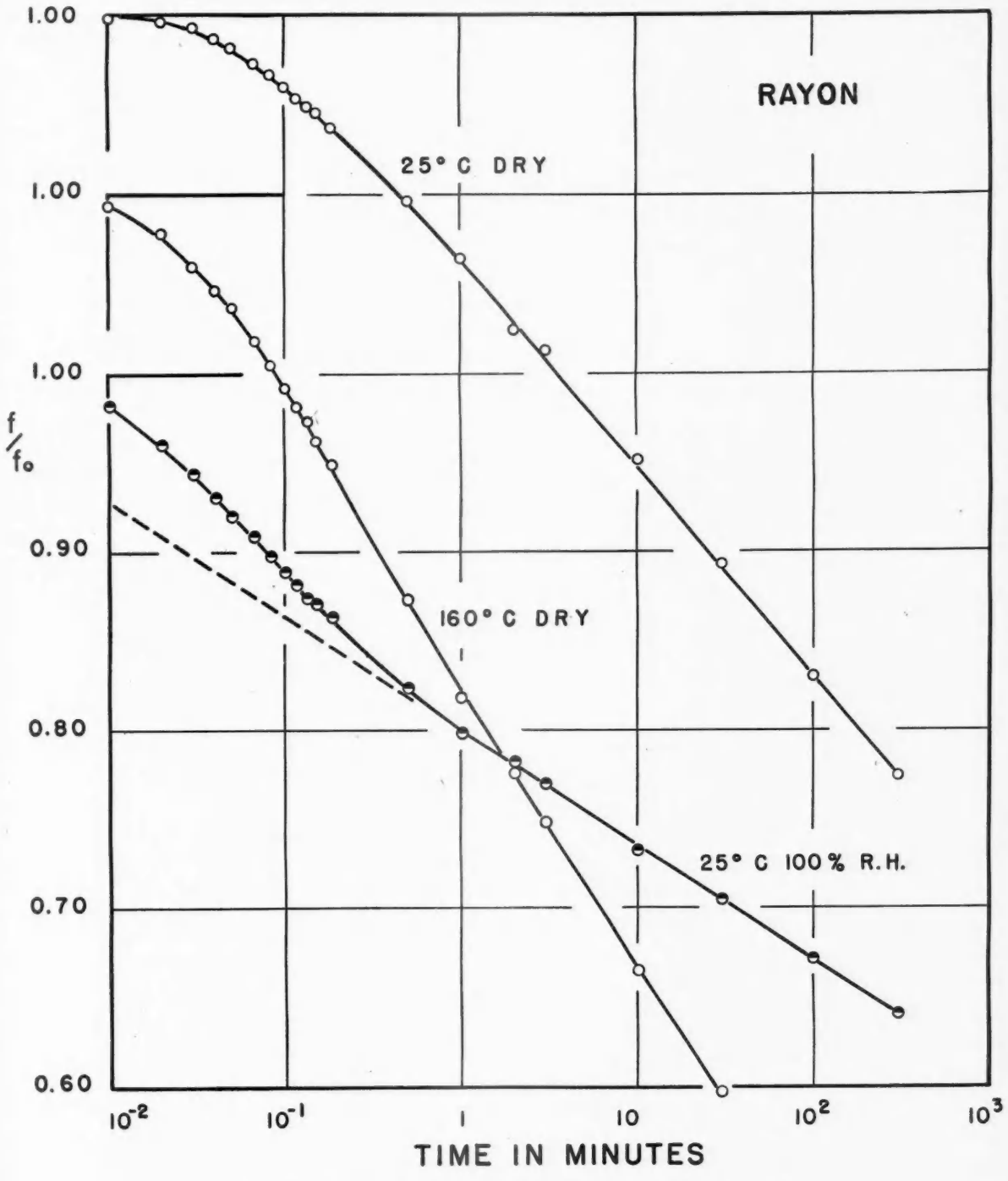

FIG. 6. *Experimental stress-relaxation curves for rayon.*

interfiber slippage should continue in the same direction after clamping when the stress is decaying. This view is supported by the fact that the rayon cord with continuous filaments showed higher rates of stress decay than the cotton cord composed of staple fiber showed. If fiber-on-fiber movement were a factor in stress decay, the opposite could logically be expected.

TABLE I. MOLECULAR CONSTANTS FOR STRESS RELAXATION OF COTTON AND RAYON CORDS

Temp. (°C)	Relative humidity	Moisture content (Dry basis)	f_0 (dynes/cm²) $\times 10^8$	$Bf_0/2$	B $\times 10^{-8}$	N $\times 10^{13}$	Nc/N	k'	G $\times 10^{10}$	A	$\Delta F^{\ddagger}$ (Calories)
				For cotton ($Nc = 3.05 \times 10^{14}$)							
25	—	Dry	15.50	16.4	2.11	5.95	5.1	8.0×10^{-15}	7.6	5.0×10^{-18}	42,400
160	—	Dry	7.75	9.2	2.37	3.66	8.3	2.5×10^{-9}	8.7	1.23×10^{-12}	51,250
110	—	Dry	7.75	11.6	2.99	3.27	9.3	4.3×10^{-11}	8.0	1.8×10^{-14}	48,450
25	—	Dry	7.75	13.2	3.41	3.70	8.2	1.24×10^{-12}	7.6	4.8×10^{-16}	39,700
25	37%	4.5%	7.75	12.6	3.25	3.88	7.9	1.11×10^{-11}	8.7	3.9×10^{-15}	38,450
25	70	6.2	7.75	12.4	3.20	3.94	7.7	2.0×10^{-11}	8.3	7.5×10^{-15}	38,100
25	100	25 (approx.)	7.75								
		For f_0'' (max)		17.2	4.44	2.84	10.7	6.9×10^{-15}	5.2	3.0×10^{-18}	42,700
		For f_0' (max)		2.0	0.52	24.40	1.2	6.2	5.2	2.3×10^{-2}	21,050
		For f_0' (min)		1.0	0.26	48.80	0.6	6.7	5.2	5.0×10^{-2}	20,600
				For rayon ($Nc = 3.08 \times 10^{14}$)							
25	—	Dry	15.64	12.4	1.58	7.96	3.9	6.0×10^{-11}	8.2	4.6×10^{-14}	37,400
160	—	Dry	7.82	7.2	1.84	4.71	6.5	9.1×10^{-7}	6.1	8.1×10^{-10}	46,300
110	—	Dry	7.82	9.2	2.35	4.17	7.4	6.4×10^{-9}	6.6	4.2×10^{-12}	44,850
25	—	Dry	7.82	10.1	2.58	4.88	6.3	9.1×10^{-10}	7.5	4.7×10^{-13}	36,050
25	37%	8.2	7.82	9.4	2.40	5.26	5.9	6.3×10^{-9}	6.9	3.8×10^{-12}	34,800
25	70	11.9	7.82	9.2	2.35	5.36	5.7	2.1×10^{-8}	6.4	1.4×10^{-11}	34,050
25	100	40 (approx.)	7.82								
		For f_0'' (max)		16.6	4.25	2.97	10.4	2.1×10^{-14}	4.9	9.9×10^{-18}	42,400
		For f_0' (max)		1.7	0.435	29.00	1.1	6.4	4.9	3.0×10^{-2}	21,300
		For f_0' (min)		1.0	0.26	49.20	0.6	6.0	4.9	4.7×10^{-2}	21,050

Effect of Initial Load

The data at 25°C for the dry cords indicate that B is not a constant but decreases as f_0 increases. This effect has also been observed with rubber, for which the function $Bf_0/2$ has been shown experimentally to be a constant independent of initial stress, so that B is inversely proportional to f_0 [25]. The significance of this relationship seems to be that the number of flow units per unit cross section increases with increasing initial stress. Obviously, in the case of cellulose, as N becomes larger it cannot increase beyond the limiting value of $N = N_C$ when the flow units are the individual cellulose chains. If we assume that at high initial loads $Bf_0/2$ has a constant value equal to that observed for the dry cords at 25°C and at initial loads of 1.0 g./Grex, we may calculate f_0^* for the case of $N = N_C$, obtaining for cotton $f_0^* = 7.9 \times 10^9$ dynes/cm² and for rayon $f_0^* = 6.1 \times 10^9$ dynes/cm². These values of f_0^* might be called the force necessary to cause all the cellulose chains to move as individual flow units.

It has been pointed out by several investigators that the theoretical or potential tensile strength of cellulose fibers, based on the force necessary to rupture the primary valence chain in cellulose, is many times the observed tensile strength [14, 19]. Various explanations have been advanced to account for this difference. Now it is recognized that the rate of elongation, or flow, in the cellulose fiber is greatly increased as the applied stress is increased. This phenomenon is probably due both to an increase in the number of shifts the flow units make per unit time interval and to an increase in the number of flow units shifting. The limiting condition will be reached when the maximum number of flow units are all under sufficient stress to move rapidly (or frequently) with respect to each other. The total stress on the fiber at this condition will then be the maximum which one might expect the fiber to bear without rupture.

It would, therefore, seem legitimate to compare the tensile strengths for the dry cotton and dry rayon cords used (which are 3.5×10^9 and 4.5×10^9 dynes/cm², respectively) with the f_0^* values of 7.9×10^9 and 6.1×10^9 dynes/cm² calculated above. In making this comparison it must be recognized that there are undoubtedly imperfections or weak spots in the macro structure of the fibers; that the observed tensile strength is measured at a finite rate of loading at which the measured value is probably lower than the maximum strength which could be obtained in a rapid test; and that in the case of a

staple cotton cord, for which the disagreement is greatest, the tested cord strength is only about half of the potential strength of the fibers. In view of these facts, the agreement between the calculated maximum strength, f_0*, and the observed breaking strengths suggests rather strongly that fiber breakage occurs by a mechanism involving slipping of the cellulose chains in the fiber rather than by rupture of those chains.

Effect of Temperature

The data of Table I show that as the temperature of the dry cord is increased the rate of relaxation with a given initial stress also increases. This seems to be simply the effect of high temperature increasing the frequency of shifts of the flow units, since the number of chains per flow unit remains about the same. $\Delta F^{\ddagger}$ increases with temperature as would be expected.

Effect of Moisture

The effect of moisture content on stress relaxation was investigated on cords which had adsorbed moisture to equilibrium at 37, 70, and 100 percent relative humidities at 25°C. Small changes in N_c/N and $\Delta F^{\ddagger}$ were observed with increasing moisture at low moisture contents. The effects observed with moisture adsorbed at 100 percent R.H. differed radically from those found at all other conditions.

Both the cotton and rayon relaxation curves at 100 percent R.H. showed a sharp change of slope at approximately 0.5 minute after the stretched cord had been clamped at a fixed strain (see Figures 5 and 6). These curves indicate that two stress-relaxation processes are initially occurring simultaneously, but that one of them is very rapid and is completed within 0.5 minute. The total stress, f, at any given time is the sum of the stress, f', due to the rapidly decaying component and the stress, f'', due to the slower component. Since $f'/f_0' = 0$ after 0.5 minute, it is possible to define f''/f_0 at any time from the straight-line portion of the curve between $t = 0.5$ minute and $t = 300$ minutes merely by extrapolating this portion of the curve back on the basis of the hyperbolic tangent decay curves observed previously.

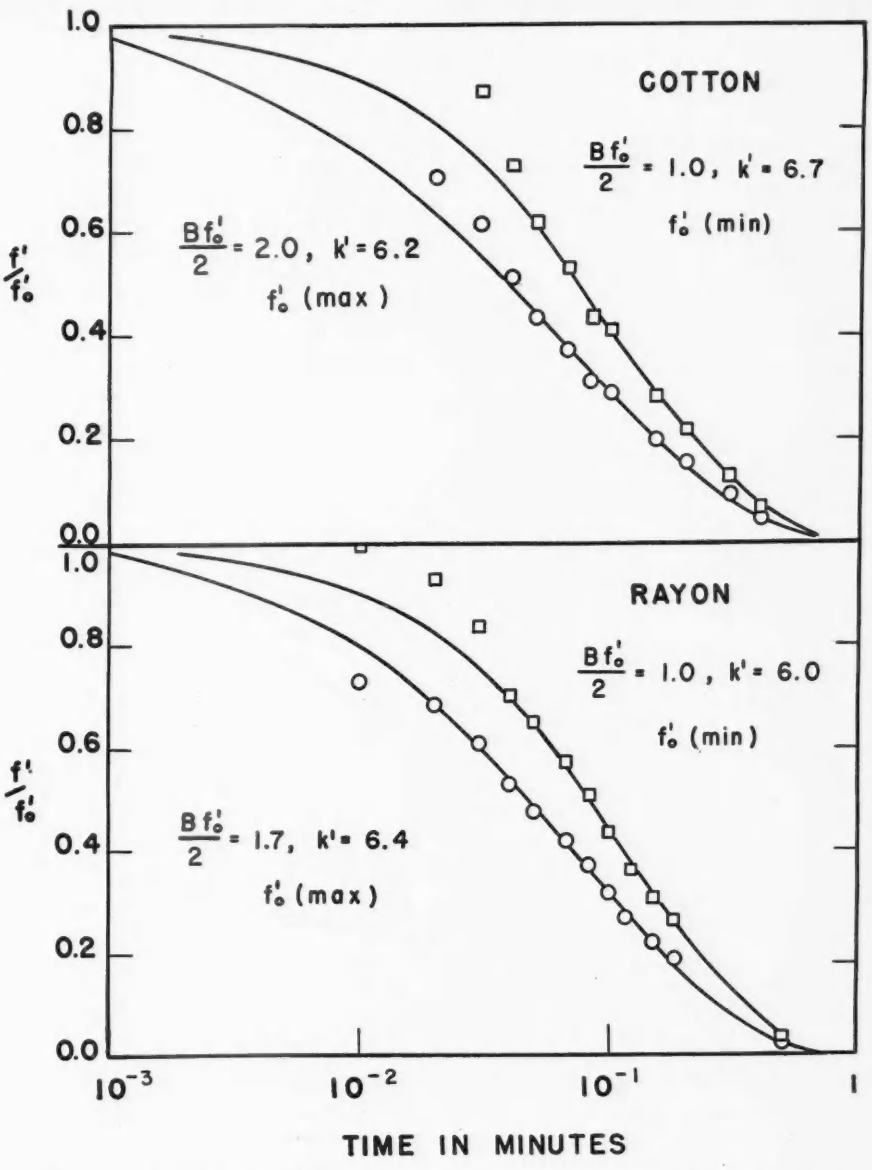

FIG. 7. *Stress-relaxation curves of rapidly decaying component in cotton and rayon at 25°C and 100% R.H. Fit of theoretical curves to experimental data.*

Subtraction of f''/f_0 from the observed values of f/f_0 gives a set of values of f'/f_0 from which the stress-decay curves of the rapid component may be calculated, providing we can assign a value to the initial stress fraction f_0'/f_0 of this component. Rigorous evaluation of this quantity is not possible in the present case, but its maximum and minimum values can be assigned. Then dividing f'/f_0 by either the maximum or minimum value gives $f'/f_0'_{(max)}$ or $f'/f_0'_{(min)}$ for various values of t, yielding data for stress-decay curves of the rapidly decaying component. These curves may be fitted to the theoretical curves for determination of the molecular constants as previously described.

Figure 7 illustrates the fit of the theoretical curves to the experimentally derived stress-relaxation data for the rapidly decaying component in cotton and rayon cords at 100 percent R.H. The fit was made for values of t greater than 0.05 minute because the observed f/f_0 at smaller values of t tended to be unreliable because of the lag in the galvanometer response to the stress signal. The calculated constants are included in Table I.

For $f_0'_{(max)}$ the number of cellulose chains per flow unit, N_C/N, is 1.2 for cotton and 1.1 for the rayon cord; for $f_0'_{(min)}$ the values are both considerably less than unity and, therefore, have no physical significance other than to indicate that the true value of f_0' must be approximately that of $f_0'_{(max)}$. The slowly decaying component contains more than 10 cellulose chains per flow unit on the average. The $\Delta F^{\ddagger}$ values for the rapid component are of the order of 21,000 calories, about half that of the slower component.

These observations on stress relaxation at high moisture contents may be explained as follows:

Much of the water taken up by the fiber is adsorbed by the hydroxyl groups of cellulose in the noncrystalline regions, where it greatly reduces the interchain bond energies and almost completely removes any stress distribution which may exist in these areas. When stress is applied to the system, a large part of the initial stress in the noncrystalline parts is first borne by the individual chains with the shortest lengths between consecutive crystallites, forcing these chains to flow through the crystallites in the direction of stress. After some flow has taken place the chain lengths between crystalline regions become more nearly equal and the stress is distributed more uniformly among the chains so that they move in multiples rather than as individuals. The resulting final stress inequality in the noncrystalline regions is thus less than that in the drier cords, in which the slippage of individual chains has been inhibited by the secondary hydrogen bonds between chains in the noncrystalline areas. Consequently, N_C/N for the final stress relaxation of the very wet cord is larger than that observed for the cord under any other condition.

Summary

1. The Tobolsky-Eyring reaction-rate theory of elasto-viscous behavior has been applied to stress relaxation at constant length for cotton and rayon cords.

2. The experimental data used in this application have been obtained by means of an apparatus employing the bonded metalectric strain gage for stress measurement without necessitating change in length of the cord. Results on stress relaxation at different conditions of humidity and temperature are reported.

3. These data have been utilized in evaluating the molecular constants in the Tobolsky-Eyring theory by assuming that the flow unit in the cellulose fiber consists of multiples of the cellobiose unit.

4. The resulting constants have permitted calculation of a theoretical tensile strength which more nearly agrees with fact than that based on primary bond strength. This suggests that the mechanism of fiber breakage is one involving slippage of the cellulose chains with respect to each other rather than the rupture of primary valence bonds.

5. For the particular case of stress relaxation at 100 percent R.H. the process has been resolved into two components, the more rapid of which appears to be the limiting case in which cellulose chains are flowing as individuals.

Acknowledgment

It is a pleasure to acknowledge the suggestions and assistance of Miss Edith Honold in many phases of the experimental part of this investigation.

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Diffusion of Water Vapor Through Textiles*

Lyman Fourt† and Milton Harris†

Abstract

One factor influencing the suitabilty of fabrics for clothing is resistance to the passage of water vapor. Measurement of the low resistances of woven fabrics requires a different technique from that for the high resistances of waterproofing and packaging materials, since the resistances are close to that of air itself, which may be used as a convenient comparison standard.

The resistance of a woven fabric depends on the kind of fiber, the thickness, and its tightness of weaving. With glass and vinyon, the resistance increases rapidly with density, showing that the main pathway through these materials is the air spaces. With cotton, viscose rayon, and wool, the tightest weaving produces resistances only four to eight times that of air, whereas those of ordinary fabrics range from two to four times that of air. This indicates that significant amounts of water vapor travel through the substance of the fibers themselves, which is confirmed by experiments on viscose film (cellophane). Nylon and cellulose acetate are intermediate. The effects of resistant fibers are seen when the fraction of the volume occupied by fiber exceeds 40 percent; below this level all fabrics are closely similar, since the low-resistance pathway through the air spaces is sufficiently available.

Background of Problem

One factor influencing the suitability of fabrics for clothing is their resistance to the passage of water vapor. This factor is important for both hot- and cold-climate clothing, but, although long recognized, does not seem to have been measured prior to the war as an intrinsic property of fabric, separate from the associated air layers. The measurements which had been made [4, 7, 14, 15] were expressed in terms of the rate at which water vapor passed through a complex system including not only the fabric but also air layers which contributed a much larger fraction of the total resistance. These results, therefore, were characteristic of the systems of measurement rather than of the fabrics themselves, although they indicated, as Gregory [7] concluded in 1930, that vapor diffusion through fabrics is independent of the rate of passage of air under pressure.

During the war, this problem was attacked by several groups of workers, among them Peirce, Rees, and Ogden [12, 13] at the Shirley Institute, Baxter at the Wool Industries Research Association [3], Goodings, Kitching, and Tucker [6, 17] at the Ontario Research Foundation, and Lee, White, and Kelso [11] at the University of Queensland. In connection with work on uniform fabrics done for the Subcommittee on Clothing under the National Research Council and the Research and Development Branch of the Office of The Quartermaster General, we also undertook measurements of this kind, the results of which are published here. The parallelism and overlapping of wartime work in different countries arose from the urgency of similar problems, and the general accordance of conclusions from the recent world-wide attack on this problem is noteworthy. The present results may, however, add certain details or points of view to the general picture.

Outside the clothing field, a great deal of work on the passage of water vapor through materials has been done in the fields of plastics, paper, packaging materials, and electrical insulation. This work has resulted in standard methods of testing for paper [16] and plastics [2]. Similar methods have been used for leather [8]. The point of view in these fields might be said to be oppositely directed from that of the field of clothing, or of physiology, however, since the concern of the packaging specialist is to prevent or reduce the passage of water vapor, whereas in clothing the desire is usually to facilitate evaporation. Thus, the order of magnitude of the

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† Harris Research Laboratories, Washington, D. C.

permeabilities in the packaging or plastics or insulation fields is very much lower than that in the textile field. A packaging material may permit the passage of only a few grams of water per square meter per 24 hours, under the usual moisture concentration gradients, whereas the evaporation from the normal skin with no active secretion of sweat is of the order of 15–25 g./m² hr. [18] and in conditions of warm environment or exertion, the evaporative cooling requirement rises to more than 100 g. water/m² hr.

Method

Definitions

This difference in direction of view and order of magnitude makes a resistance unit appropriate for the description of clothing fabrics. The natural standard of comparison in this field is the resistance of ideal still air to the passage of water vapor by diffusion, since this kind of resistance plays a significant rôle both in measuring systems and in the biophysics of clothing. As a unit, therefore, we can take the resistance of one centimeter of ideal still air. In order to compare different fabrics, it is convenient to calculate the relative resistance of each, as its resistance (in terms of distance in an air column) per unit thickness. This thickness should be measured at a low pressure: 0.1 lb./in² was used in the present work, but an even lower pressure would be appropriate, especially for compressible materials.

The total resistance, R, of a system is given in equivalent centimeters of still air, in terms of Q, the grams of water vapor passing; D, the diffusion coefficient; ΔC, the difference in vapor concentration; A, the area in cm²; and t, the time in seconds, by the equation

$$R = \frac{1}{Q} D(\Delta C) A_t. \tag{1}$$

The diffusion coefficient, D, varies with the average absolute temperature, T, and the barometric pressure, p (mm. Hg), according to the relation [9]

$$D = 0.220 \left(\frac{T}{273}\right)^{1.75} \frac{760}{p}. \tag{2}$$

With sufficient practical accuracy, this may be replaced between 0° and 50°C by

$$D = 0.220 + 0.00147\, m, \tag{3}$$

where m is the temperature (°C).

The concentration difference, ΔC, in g./cm³ can be obtained from the relative humidities H_1 and H_2, the absolute temperatures T_1 and T_2, and the corresponding saturation vapor pressures p_1 and p_2 on each side of the resistance as

$$\Delta C = \frac{18}{22{,}400} \times \frac{273}{760} \left(\frac{p_1 H_1}{T_1} - \frac{p_2 H_2}{T_2}\right). \tag{4}$$

For one temperature, this becomes

$$\Delta C = 2.89 \times 10^{-4} \times \Delta p / T, \tag{5}$$

where Δp is the difference in actual vapor pressures.

To find the permeability under any conditions, the equation can be solved for Q/At and evaluated from the appropriate values of D, ΔC, and R:

$$\frac{Q}{A_t} = \frac{\Delta C}{R} \times D. \tag{6}$$

The preceding equations (1 through 6) have dealt with the total resistance or over-all permeability of complete systems. The intrinsic resistance of a fabric is defined as the difference in total resistance caused by an additional layer of fabric. The measurement of the intrinsic resistance of a fabric can be made by comparing the total resistance of two systems containing fabric, with one having more layers than the other. All the air spaces in the two systems must be made equal or measured and accounted for.

Preparation of Sample

Usually at least three test systems are prepared for each fabric, with 1, 2, and 3 layers of the fabric, respectively. The simplest procedure for multiple layers is to place each layer in direct contact with the next, avoiding wrinkles, which would introduce additional air-space resistance. Alternatively, cloth layers can be separated but the separating air layers must be accounted for in estimating the intrinsic resistance, and should be kept small, to avoid convection disturbances. An experimental test for the absence of convection is that the total resistance should be increased by exactly the amount of any increase in the space between the layers.

The area under test (17 cm² has been used) is defined by sealing the fabric with wax (50 percent beeswax, 50 percent paraffin) between metal rings. Care must be exercised not to apply an excess of wax too close to the opening to avoid flow into the open space. It is considered better to have the wax reach not quite to the opening.

Measurement of Diffusion

The quantities required for calculation by equation (1) can be obtained by either of two methods.

A. Absorption Cup Method. The assembly of fabric and metal rings is sealed to small dishes containing a granular drying agent. The dishes are inverted so that the drying agent is in contact with the fabric, minimizing the path of the water vapor. The inverted dishes are placed on a wide mesh wire rack in a uniform current of conditioned air (70°F, 65 percent R.H., 200 ft./min. has been used). Drierite (anhydrous $CaSO_4$) and anhydrone ($MgClO_4$) are satisfactory drying agents; the $MgClO_4$ lasts longer, but has a tendency to become wet and stick to the cloth unless mixed at frequent intervals. Weighings are made at uniform intervals of $\frac{1}{2}$ to 1 hour, and the drying agent is mixed at each weighing, since otherwise the resistance tends to increase as the layers next to the fabric become saturated. The rate of gain in weight becomes constant after the first half hour, and remains constant for 2 hours or more.

B. Evaporation Procedure. The layers of cloth are sealed into metal rings in the same manner as in the absorption procedure. The opening is the same as the average inside diameter of the straight-sided, cylindrical vessel used (Kimble 50 × 35 mm. crystallizing dishes) in order to secure linear diffusion inside the cup. A special ring, with a shoulder recessed to fit the edge of the dish, is an aid to centering. Since glass vessels vary somewhat in diameter, and in contour across the bottom, each must be individually calibrated. This can be done by finding the weight of water required to fill it flush with a flat glass plate lying across the rim. From the volume, and the area of the top, the volume required to fill the cup to any given distance from the brim can be computed. A convenient distance, including the thickness of the metal ring below the cloth, is 1 cm., and, unless otherwise stated, this standard separation is used, although the distance may be as much as 2.5 cm. In this respect the method differs from that of the American Society for Testing Materials [2] or TAPPI [16], in which a space of 2.5 cm. (or greater) is recommended. Variation in the space beneath the cloth causes an equal variation in the total resistance, within a range up to about 2.5 cm. At greater spacings, the total resistance does not increase by the full amount of the extra space; in fact, it increases only slowly with increasing separation, presumably because of convection disturbances, as suggested by Langmuir and Schaefer [10]. If the amount of evaporation is sufficient to lower the water level in the interval between weighings, the average spacing should be used in accounting for the air space underneath the fabric. The dishes are exposed in a uniform air stream and weighed at intervals of about an hour. The apparent resistance may be high for the first few readings on account of the absorption of moisture by the cloth, but a steady rate is soon reached. For the purpose of calculating the resistance, it is assumed that evaporation is taking place at room temperature. The actual temperature of the evaporating surface is somewhat below room temperature, and hence the actual difference in concentration between saturated vapor at the water surface and the air of the room is less than the assumed, which implies that the actual resistance is somewhat less than the calculated resistance. Another source of error in the evaporation procedure is the tendency of the fabrics to sag. Uniformity can be established by gentle pressure in the centers of the circles of fabric.

Results

Consistency and Precision

Typical results of diffusion measurements are shown in Figure 1. In this experiment, each number of layers of fabric was set up in duplicate, and the two observations are plotted as coinciding or adjacent points. The general agreement of the two

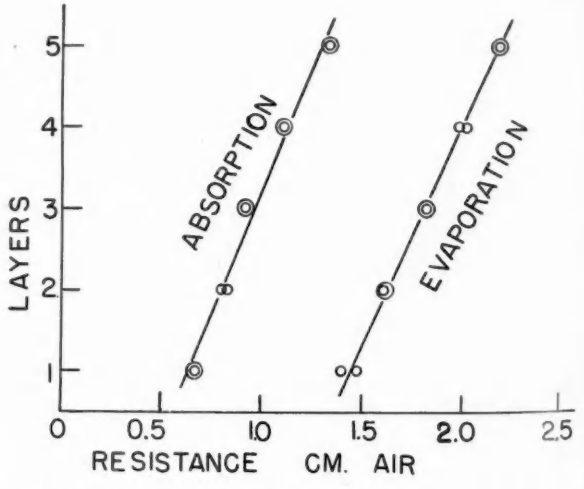

FIG. 1. *Typical results of diffusion measurements of fabrics (cotton sateen).*

TABLE I.

	Evaporation method			Absorption method		
	Resistance (cm. air)	Gradient (% R.H.)	Range (% R.H.)	Resistance (cm. air)	Gradient (% R.H.)	Range (% R.H.)
Air outside (by difference)	0.34	7.7	65.0– 72.7	0.34	25.1	65.0–39.9
Fabric	0.20	4.5	72.7– 77.2	0.20	14.8	39.9–25.1
Air inside	1.00	22.8	77.2–100.0	0.34	25.1	25.1– 0.0
Total	1.54	35.0	65.0–100.0	0.88	65.0	65.0– 0.0

procedures, evaporation and absorption, is also seen. The average increment in resistance per layer—that is, the intrinsic resistance of the fabric—is the average slope of such plots.

Since the intrinsic resistance of fabrics is small, often only 0.1 to 0.2 cm., in comparison with the total resistance, the percent precision of the measurement of intrinsic resistance is less than that of total resistance. The additional resistance of air layers, in the air current above the fabric and between the fabric and the point of absorption or evaporation, has been observed to be 0.80 cm. with drierite (method A), 0.66 cm. with anhydrone (method A) and 1.34 cm. with water 1 cm. below the fabric (method B). In a series of 30 measurements of fabrics, the average range of variation in the comparison of 1 and 2 layers, and 2 and 3 layers, is 0.031 cm. on either side of the average (methods A and B together). This indicates that the measurements are significant in terms of tenths of centimeters, but that the hundredths figure is not fully significant.

Comparison of Absorption and Evaporation Methods

The average difference between the resistance, as measured by method A, in which the fabric is relatively dry, and method B, in which it is relatively moist, is 0.05 cm. air in the 34 comparisons on identical specimens in Table I. In 19 of the comparisons, the evaporation method indicated a resistance on the average 0.06 cm. of air greater than the absorption method. In the 15 comparisons in which the absorption method showed the same or a larger resistance, the average difference was 0.03 cm. of air. This indicates that within the precision of the present technique, there is little difference between the results obtained by the two methods over a wide range of woven and knitted fabrics, although a small systematic error, which would be expected from consideration of temperature effects, may show up in the results of the evaporation method. However, for very thick or dense fabrics a difference may be found. The reasons for the nearly complete absence of difference between the two methods for knit or woven textiles, in contrast to the frequent observation of such differences for packaging materials, paper products, and cellophane, are made clear from a consideration of the actual levels of relative humidity in the specimen layers, as suggested by Goodings [17].

Effect of the Level of Relative Humidity in the Specimen

The total gradient of relative humidity in each type of diffusion system is determined by the conditions at the ends of the columns—using the ASTM standard textile testing atmosphere for one end of the column, this gradient is from 65 percent to 0 percent R.H. for absorption, and from 65 percent to 100 percent R.H. for evaporation. However, this total gradient must be divided in proportion to the three individual resistances: (1) of the air layers outside the diffusion cups; (2) of the specimen layers; and (3) of the air layers inside the cup. Thus for a typical fabric layer with a resistance of 0.2 cm. the divisions of the resistances and of the humidity gradients are those listed in Table I.

This type of calculation shows that in the systems in which the fabric contributes only a small part of the total resistance, the fabric is in an intermediate zone of humidity, between 80 and 20 percent R.H., whichever method is used.

When, however, the specimen contributes a large proportion of the total resistance, a larger portion of the total gradient is contained in the specimen layers, and its surface layers may be in more extreme conditions of relative humidity. That such conditions do affect the apparent resistance is shown by an experiment with plain transparent cellophane (not waterproofed), 0.004 cm. thick, density 1.31

TABLE II.

	Evaporation method			Absorption method		
	Resistance (cm. air)	Gradient (% R.H.)	Range (% R.H.)	Resistance (cm. air)	Gradient (% R.H.)	Range (% R.H.)
Air outside	0.34	7.9	65.0– 72.9	0.34	4.0	65–61
Cellophane	0.16	3.7	72.9– 76.6	4.8	57.0	61– 4
Air inside	1.00	23.4	76.6–100.0	0.34	4.0	4– 0
Total	1.50	35.0	65.0–100.0	5.48	65.0	65– 0

TABLE III.

Fabric	Fiber vol. (%)	Weight (oz./yd²)	Thickness at 0.1 lb./in² (in.)	Air permeability (ft³/ft² min. at 0.5 in. w.g.)	Method*	Intrinsic resistance (cm. air)	Relative resistance (x air)
Cottons							
Knit undershirt	13	4.2	0.028	223	A	0.12	1.7
					B	.07	1.0
Balloon cloth	25	2.0	.007	100	A	.047	2.61
					B	.047	2.61
Bedford cord (Navy "jungle" cloth)†	27	13.6	.043	6	A	.37	3.4
					B	.41	3.8
Herringbone twill, OD 7	27	8.1	.026	13	A	.11	1.65
					B	.13	2.19
Shirley cloth†	33	9.6	.025	1	A	.22	3.5
					B	.27	4.3
Poplin, 5-oz.	33	6.2	.016	6	A	.09	2.2
					B	.10	2.46
Sateen	34	9.7	.025	6	A	.165	2.62
					B	.185	2.94
Uniform twill, 8.2-oz.	35	8.2	.022	12	A	.23	4.2
					B	—	—
Byrd cloth	37	5.2	.013	3	A	.11	3.3
					B	—	—
L19 Shirley†	41	7.4	.016	1	A	.14	3.4
					B	.18	4.4
J. P. Stevens 1650	48	7.9	.015	1	A	.14	3.8
					B	.17	4.6
Vinyon							
Knit	23	4.8	.021	256	A	.090	1.7
					B	.070	1.3
2056	29	4.2	.014	16	A	.150	4.2
					B	.184	5.2
2069	30	4.7	.013	294	A	.123	3.7
					B	.141	4.3
Sail cloth, light	35	2.55	.025	1	A	.464	7.3
					B	.473	7.4
2056	36	4.0	.011	17	A	.18	6.6
					B	—	—
Filter No. 8	38	14.8	.033	9	A	.34	4.0
					B	.38	4.5
Sail cloth, medium	41	5.35	.013	1	A	.43	13.0
					B	.40	12.2
Filter No. 19	44	14.3	.031	9	A	.46	5.9
					B	.42	5.4
Filter No. 1	47	13.7	.028	1	A	.68	9.6
					B	.67	9.4
1802	49	3.5	0.007	1	A	.28	15.4
					B	0.35	19.8

TABLE III.—*Continued*

Fabric	Fiber vol. (%)	Weight (oz./yd²)	Thickness at 0.1 lb./in² (in.)	Air permeability (ft³/ft² min. at 0.5 in. w.g.)	Method*	Intrinsic resistance (cm. air)	Relative resistance (x air)
Glass							
CSS-28 extracted	16.5	26.5	0.083	12	A	0.30	1.42
					B	.33	1.56
CSS-20	20.4	24.3	.057	8.6	A	.38	2.62
					B	.365	2.52
WB 0046	21.6	—	.058	—	A	.521	3.54
					B	.438	2.98
WB 0111	22.0	—	.074	—	A	.423	3.00
					B	.393	2.78
CSS-28, original	22.4	27.6	.064	8.5	A	.42	2.58
					B	.46	2.82
ECC-11-138	24.7	6.7	.014	6.0	A	.22	6.34
					B	.20	5.56
ECC-11-128	32.2	7.4	.012	8.0	A	.42	13.9
					B	.33	10.7
No designation	38.8	9.6	.013	—	A	.33	8.0
					B	.31	7.5
Wool							
Wool serge, 16 oz.	25	10.4	.038	12	A	.18	1.9
					B	.26	2.7
Serge, 45% viscose, 55% wool	31	11.5	.033	10	A	.20	2.4
					B	.18	2.1
Wool felt	18	19.1	.103	24	A	.37	1.4
					B	.67	2.6
Wool felt	17	31.3	.184	16	A	.80	1.7
					B	1.10	2.4
Nylon							
Knit	31	6.5	.025	108	A	.16	2.5
					B	.15	2.3
3-oz. twill	46	3.1	.008	21	A	.15	7.5
					B	.20	10.0
5-oz. twill	49	4.6	.011	20	A	.20	7.1
					B	.18	6.4
Mountain tent, calendered	60	3.2	0.006	2	A	.32	20.0
					B	0.33	20.6

* Method A = absorption; method B = evaporation.
† Indicates water-repellent treatment on fabric.

g./cm. This was used in its original state. The intrinsic resistance by the evaporation method was 0.16 cm. air; by the absorption method, however, 4.8 cm. air. The gradient picture for one layer of this material is shown by Table II.

Thus, even in the least severe situation—when there is only one layer—the inner surface of this layer is in equilibrium with air at only 4 percent R.H.

Effect of Fiber Content and Density of Structure

The results of measurements on a variety of fabrics are shown in Table III. It will be noted that in general the two methods are in close agreement.

In order to compare the effect of fiber composition, whether of a cotton, wool, glass, etc., on a uniform basis, the fraction of the total volume taken up by fiber has been computed. This is found from the apparent density by dividing by the density of the fiber substance, using the following values: cotton or viscose rayon, 1.55; wool, 1.33; nylon, 1.15; glass, 2.55; vinyon, 1.35. This procedure enables us to compare the effect of density of structure for the different fibers, as shown graphically in Figure 2.

With glass and vinyon, the resistance increases rapidly with density to twenty times that of air,

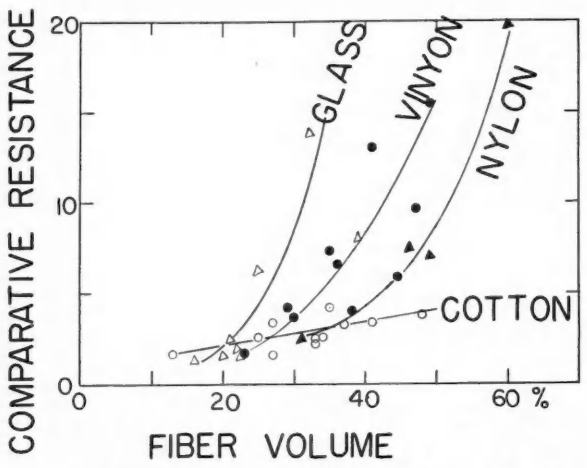

FIG. 2. *Comparative resistances of fibers of different densities of structure and fiber content.*

showing that the main pathway through these materials is the air spaces. With cotton, the tightest weaving produces resistances only four times that of air, whereas for most cottons the range is nearer two times the resistance of air. This indicates that significant amounts of water vapor travel through the substance of the fibers themselves, as is confirmed by the experiment on cellophane discussed in connection with level of humidity. Nylon and cellulose acetate are intermediate. The effects of resistant fibers are seen when the volume occupied by the fiber exceeds 30–40 percent; below this level all fabrics are closely similar, since the low-resistance pathway through air spaces is sufficiently available.

Effect of Air Permeability

The permeability to air under pressure is indicated in Table I, showing a wide range for each kind of fiber. Under diffusion conditions, however, with no penetration of the fabric by masses of moving air, the effect of air permeability is manifest only through the related effect of density, which in turn depends on the kind of fiber. For the absorbent, low-resistance fibers such as cotton, wool, and viscose rayon, the effect of air permeability on vapor permeability or resistance is minor, as Gregory [7] and Sale and Hedrick [14] have shown.

There is an upper limit to the air permeability of fabrics suitable for test by these methods, although it is above 300 $ft^3/ft^2/min./0.5$ in. water. Evaporation trials on a mosquito net (permeability 1300) showed a total resistance of less than 1.0 cm., indicating penetration by air currents.

Effect of Water-Repellent Treatments

Data on cottons treated with water-repellent finishes are included in Table III, and indicate that these finishes do not affect the vapor resistance of these fabrics within the limits of precision of these tests.

Discussion

Stability of Diffusion Column and Validity of Methods

Consideration of the evaporation method shows that the vertical diffusion column employed in it is gravitationally unstable, since the density of moist air is less than that of dry air, unless the effects of increased water-vapor content at the base of the column are balanced by decreased temperature. This may account for the fact, noted by numerous observers [1, 10, 17], that the contribution of an air space to the total resistance levels off, under ordinary conditions of experimentation, at about 2.5 cm. Goodings [6] has made use of two different spacings inside the evaporation cups. This amounts to determining the combined effect of the diffusion constant and the concentration gradient experimentally, instead of calculating from temperature, relative humidity, and the tabulated values of D. An interlaboratory comparison of these methods gave the results shown in Table IV. These show, in most instances, very close agreement between the absorption method and Goodings' evaporation method with experimental determination of the constants, and general agreement with the evaporation method as outlined here, which uses calculated constants and an assumed temperature of the evaporating surface equal to room temperature. However, the point noted in the description of the method—that the actual resistance should be somewhat smaller than

TABLE IV.

Fabric	Thickness (cm.)	Resistance per layer, cm. of air: Goodings (evaporation)	Method A (absorption)	Method B (evaporation)
Balloon cloth	0.018	0.04	0.05	0.07
5-oz. poplin, wind-resistant	0.039	0.09	0.09	0.14
Shirley cloth	0.064	0.23	0.22	0.27
Bedford cord (Navy "jungle" cloth)	0.109	0.30	0.37	0.41

the resistance calculated for the evaporative method —is confirmed.

Significance in Choice of Clothing

These results were obtained during the war primarily with reference to the suitability of tightly woven cotton fabrics for jungle clothing. Such fabrics were desired for two reasons: (1) possible protection against mosquito bites, and (2) the finding that thin fabrics, even though tightly woven, imposed less heat stress than thicker, somewhat more porous fabrics. Although resistance to diffusion of water vapor is by no means the only factor in this choice, the results give a clear-cut answer to the question, indicating that cotton (and viscose rayon or wool) at even the limit of weaving density does not block the passage of water vapor. However, if less absorbent fibers, such as nylon or acetate, are being used, the higher-density fabrics may impose an unusual burden on evaporative cooling.

It should be pointed out that the factors of thickness and air permeability are also involved in the choice of hot-climate clothing. These have been the subjects of additional study.

Not only the clothing itself limits evaporation, but also the layers of relatively still air in the clothing or surrounding it and the body. Gagge [5] has found that the maximum evaporation rate from the body, without clothing, under conditions of low air movement (17 ft. per min.) is 2.85 kg. cal./m²hr. (mm. vapor pressure difference), from which the resistance to evaporation is equivalent to a layer of ideal still air 1.86 cm. thick. This is, of course, reduced by air movement, but in comparison with this resistance under "telephone-booth conditions" the resistances of the fabrics in most clothing are small.

In real situations, if the amount of clothing remains constant, a man adjusts to the resistance of his clothing in either of two ways: (1) by increasing the wetted area of the skin, as described by Gagge [5], or (2) by saturating the clothing with sweat. In the second case, resistance of the clothing to passage of water vapor is no longer a factor. Thus, the relatively small differences in the vapor resistances of different fabrics have little effect on the efficiency of active men, but under relatively quiet conditions these differences, especially as amplified by multiple layers in clothing, can probably be noticed in terms of comfort.

Acknowledgment

The authors wish to acknowledge the assistance of Miss Polly Logan, Miss Mary Ellen Parish, and Miss Katharine Fisk in performing the measurements involved in this work.

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Investigation of Fine Structure of Freely Coagulated Viscose Fibers by Means of Optical and Electron Microscope*

M. Horio, K. Kobayashi, and T. Kondo

Institute of Fiber-Chemistry, Faculty of Engineering, Kyoto Imperial University, Kyoto, Japan

1. Introduction

Cross sections of viscose fibers have been studied microscopically by numerous research workers. In the earlier studies interest was directed to the shape of the cross sections, which depends upon the conditions of the spinning dope, and especially upon the composition of the coagulating bath.

Preston [1] was the first to report the inhomogeneous construction of a cross section by the aid of metachromatic dyeing. He distinguished the "skin," where an axial orientation of micelles could be assumed, from the unoriented interior. He connected further the occurrence of the skin with the streaming condition of the viscose during spinning, which was extruded through the spinneret holes into the bath and drawn by a godet wheel. Van Bergen and Bredee [2], Schramek and Zehmisch [3], Löbering [4], and others came to a similar conclusion. By the aid of metachromatic dyeing and precipitation of minute metallic crystals of gold and silver in the fiber tissue, Ohara [5] presumed that the skin itself is composed of two or more layers which differ in density.

One of the present authors (K. K. [6]) showed, however, that the tangential orientation of the micelles could happen even if the viscose was coagulated freely without being drawn or stretched, and pointed out the inconsistency of Preston's opinion. Detailed observations of the structure of the freely coagulated fibers led us to the conclusion that their construction does not differ essentially from that of the fibers spun-drawn and stretched in the usual way. The elimination of drafting gave a larger fiber dimension with easier sectioning and observation. Thus the unstretched spun fibers often had advantage over the ordinary spun fibers for our studies since they gave more detailed and distinct figures. Drafting and stretching during spinning are effective only in the sense that they bring about a decrease in the thickness of the skin and central layer of the fibers in almost the same proportion and introduce the axial orientation of the structural units.

In the present paper we want to deal with the optical and electron microscopic structure of freely coagulated viscose fibers and to discuss the phenomena which take place when viscose is spun. These remarkable phenomena, in spite of their great importance, have not been represented strikingly by any authors, so far as we are aware.†

2. Optical Microscopic Observations

Samples of the freely coagulated fibers were prepared in the following way:

The viscose, consisting of cellulose 8 percent and total alkali as caustic soda 7 percent and having an ammonium chloride number 10–14, was extruded from an ordinary spinneret into a coagulating bath containing sulfuric acid 130 g./l., sodium sulfate 250 g./l., and zinc sulfate 5 g./l., and was coagulated freely without being drawn and stretched. The desulfurized and washed filaments were embedded in paraffin in the usual way and sectioned. In order

* This paper was submitted for publication in TEXTILE RESEARCH JOURNAL directly by Dr. Horio. Another paper by him and one by K. Kobayashi appeared in the October, 1946, issue.

† A paper by Morehead and Sisson on "Skin Effect in Viscose Rayon" appeared in TEXTILE RESEARCH JOURNAL **15**, 443 (1945). This was probably not available in Japan when Professor Horio was preparing this report.—*Editor*

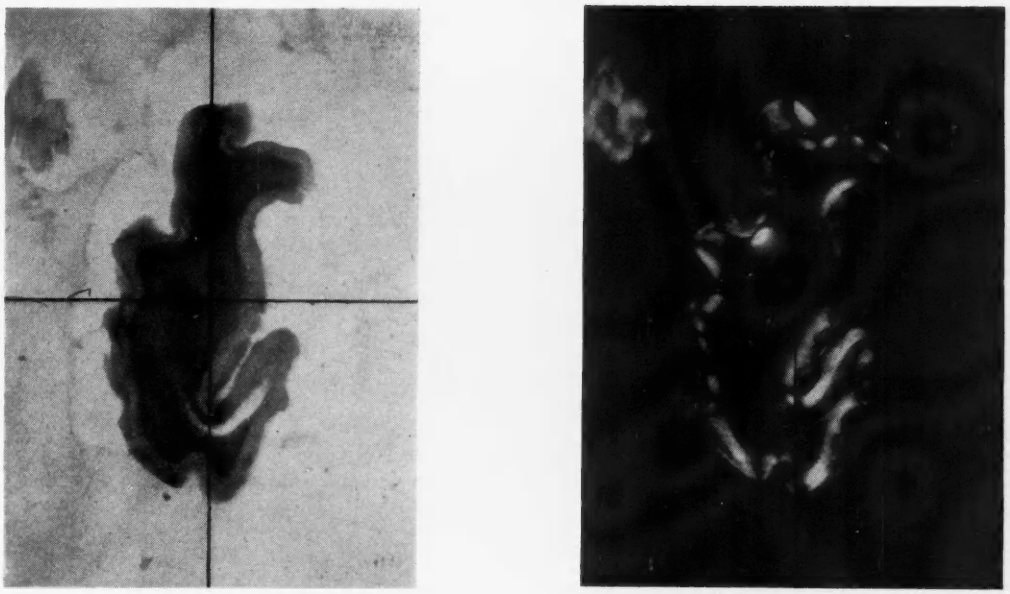

FIG. 1.* *Cross section of an unstretched coagulated viscose fiber (600 ×).*

A (left)—Dyed with Oxamine Blue 4RX and observed by direct light. Immediately after dyeing the skin was blue, and the center was a darker blue. If the dyed sections were washed with water, the dye in the center washed out rapidly, and the color faded, whereas the blue in the skin was fixed more strongly.

B (right)—The same section observed by polarized light between crossed Nicols. Diagonally positioned portions of the skin are brightened. The skin is composed of three layers: a dark one inserted between two bright ones.

* Figures 1–4 are optical photomicrographs, and Figures 5–15 are electron photomicrographs.

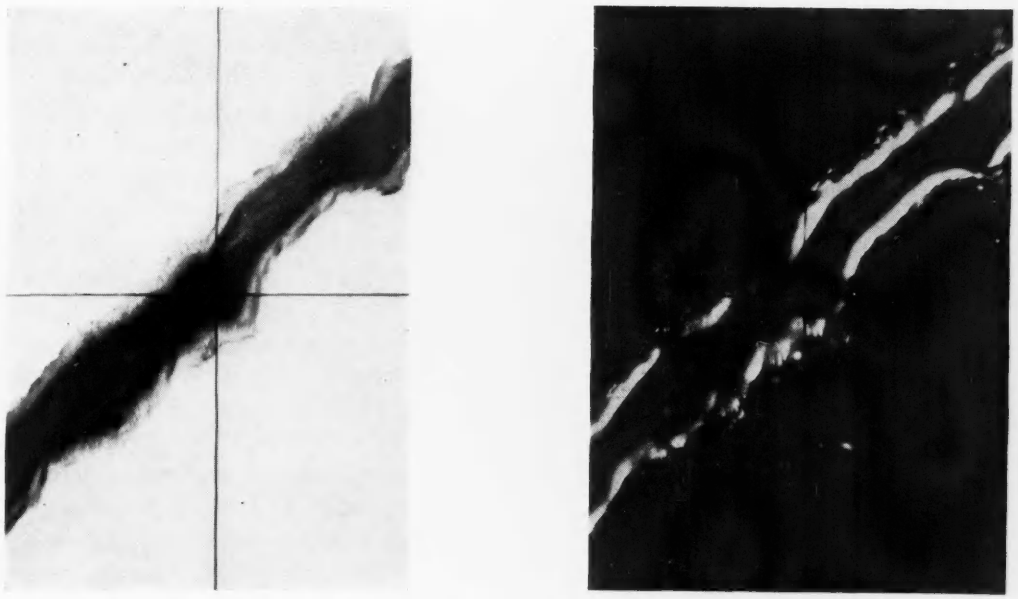

FIG. 2. *Longitudinal (radial) section of an unstretched coagulated viscose fiber (600 ×).*

A (left)—Observed by direct light.

B (right)—Observed by polarized light between crossed Nicols. The explanation for Fig. 1 applies here also.

that the metachromatic behavior might be examined, the sections were dyed with Oxamine Blue 4RX.

Figures 1 and 2 show the cross and longitudinal (radial) sections as observed with both direct (Figures 1a and 2a) and polarized (Figures 1b and 2b) light. In the figures one can see a very marked skin. The figures of the sections observed by polarized light suggest the bare possibility that the structural units are oriented tangentially.* If we recall that the fibers were prepared so as to exclude the possibility of drafting and stretching, we cannot attribute the occurrence of the skin to the hydrodynamic orientation of the micelles as concluded by Preston [1] and others.† Furthermore, even if we draw the filaments with a reel and stretch them by the aid of guides between the surface of the bath and the reel, the proportion of the skin does not change perceptibly. The occurrence of the skin is, therefore, conditioned neither by the streaming of the viscose nor by the stretching of the filaments, but by the chemical and colloid-chemical changes to which the viscose has been subjected in the coagulating bath.‡

If the photomicrographs taken with polarized light (Figures 1b and 2b) are examined carefully, it can be seen that the skin itself consists of three layers. Between two oriented layers there exists an isotropic one. It is impossible to estimate the real thickness of these layers by such optical microscopic observations, but it can be anticipated that the outermost layer would be thinner than the inner oriented layer and that a very thin isotropic layer exists between them.

Within the core of the filament we can perceive several layers which succeed one another and differ in density and orientation of the micelles. Such a succession of layers can be observed more easily if the viscose is spread on a piece of glass plate and subjected to the action of the usual spinning bath. Figure 3 shows the cross section of a film thus obtained, observed by direct and polarized light.

We believe that a similar succession of layers could occur also in viscose fibers, spun in the usual way, but owing to drafting each layer is attenuated, and the structure, especially that of the interior, would be greatly disturbed by such mechanical treatments.

It is very difficult, as in most cases of other Liesegang phenomena, to explain how this layer succession occurs. Later, we want to discuss this problem in detail. Mention must be made at this point about another important result. If the viscose is coagulated with a solution of neutral salts such as sodium or ammonium sulfate, or even if the coagulated mass is, in addition, subjected to the action of sulfuric acid, such a layer succession as described above cannot be seen at all. The cooperative actions of the salts and the acid seem to be of essential importance for the occurrence of the periodic coagulation. Thus, the period of the succession depends not only upon the conditions of the viscose under observation but also upon the composition of the coagulating bath. The favorable degree of ripening of viscose is 8–15 in ammonium chloride number. The content of sulfuric acid in the bath, which is one of the most important factors, must be in the vicinity of 100 g./l. If we increase the concentration of the acid, the pitch of the succession tends to be decreased.

A more important optical microscopic method, by the aid of which the submicroscopic structure of the skin and inner layer can be analyzed, is the dark-field method. Figure 4 shows a cross section of the freely coagulated viscose fiber observed with dark-field illumination.

From the figure we would be able to predict that the skin, in spite of higher density, contains many minute bubbles, whose shapes and dimensions, however, we cannot estimate precisely. On the other hand, the center is so loosened that it is an optical void.

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* If the skin were produced by the paraboloidal velocity gradient within each hole of the spinneret, as was concluded by van Bergen and Bredee [2], and Schramek and Zehmisch [3], then the orientation should not be tangential, but axial. Furthermore, the distinctly bordered discontinuous transition from the skin to the center, which can be seen in the figures, cannot be brought about by the streaming conditions.

† According to our recent study of the hydrodynamic properties of viscose (cellulose 8 percent, total alkali 6 percent, made from aged alkali cellulose), the Hagen-Poiseuille's law is valid practically up to the shearing stress of 10^4 dynes/cm^2. At 10^5 dynes/cm^2—that is, the shearing stress which predominates at the extrusion of viscose from a hole of the spinneret—slight indication of structural flow comes gradually in sight. The data of Philippoff and Krüger [7], covering a wide range of conditions of preparing the viscose, show also on the whole the characteristics of the Newton liquid up to 10^4 dynes/cm^2. At 10^5 dynes/cm^2 the structural flow is the more obvious for the more viscous viscose.

‡ The viscose was hung down in a fine stream from the point of a fine glass rod upon the surface of the coagulating bath and was subjected to its action, and yet the skin was formed as before.

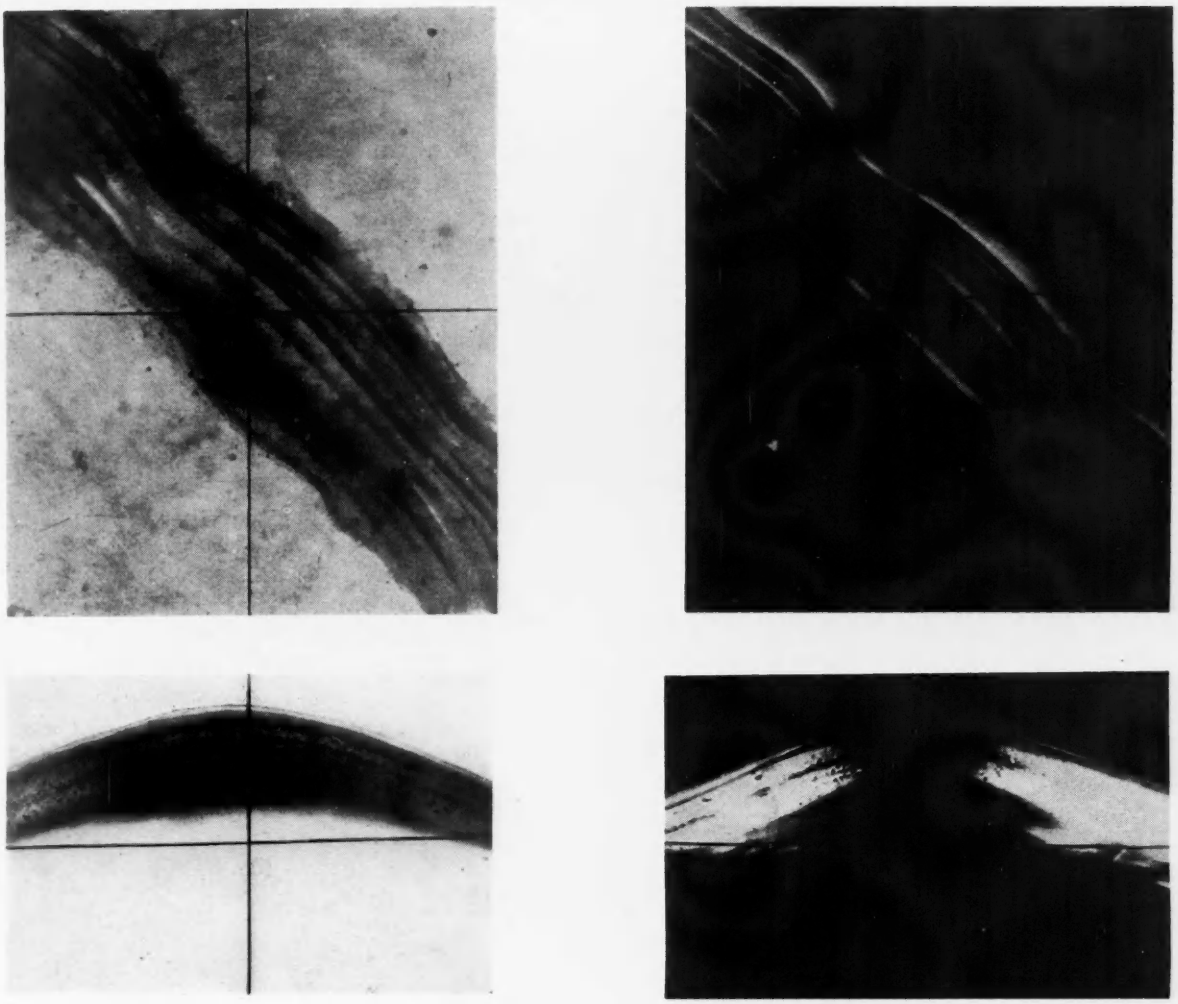

FIG. 3. *Cross section of viscose film coagulated by a bath containing sulfuric acid only (upper), and by a bath containing sulfuric acid and sodium sulfate (lower) (600 ×).*

A (left, upper and lower)—Dyed with Oxamine Blue 4RX and observed by direct light.

B (right, upper and lower)—Positioned diagonally and observed by polarized light between crossed Nicols. We see a succession of several layers. The film, coagulated by sulfuric acid only, contains a larger number of layers, while the film coagulated by a bath composed of sulfuric acid and sulfate shows a structure similar to that of the freely coagulated fibers (cf. *Figs. 1 and 2*). *Layers which appear darker-colored in direct light brighten under polarized light. This may be due to the fact that isotropic layers are not compact enough to take up sufficient coloring.*

3. Electron Microscopic Observations

As mentioned above, many interesting and important characteristics of the skin and core are of submicroscopic nature. We can of course picture the submicroscopic structure by the aid of the results of polarized light and dark-field observations to some extent, but these indirect methods give us only broad views. Overrating the results thus obtained often leads to faulty conclusions.

In order to obtain the figures of submicroscopic structure of the freely coagulated viscose fibers we employed an electron microscope of the electrostatic type. The magnifying power of the apparatus is 1,000 times, but its resolving power is 5–10 mμ for our samples. This resolving power is sufficient to analyze and interpret the submicroscopic structure which has not been revealed optically. As will be described later in detail, we adopted intentionally the

above-mentioned lower magnification,* while we endeavored to increase the resolving power as much as possible.

Many electron microscopic observations of fibrous materials have been made with disintegrated samples of fibers [8]. The purpose of our study was, however, to extend and develop our optical observations to electron optical ones. Therefore, we wanted to employ the sectioned samples of fibers, without rupturing their original structure, as in the cases of optical microscopy. The way in which this was accomplished will be disclosed later in detail.

Figure 5 shows a cross section of a freely coagulated viscose fiber, observed with the electron microscope. At a glance we see several successive layers which differ in electron transmissibility. The outermost layer is very thin and is characteristic in the respect that electrons cannot penetrate this layer at all under the present experimental conditions. This suggests that this layer has the highest density. We shall call it the "epidermis," in analogy to animal and plant tissues. Just below the epidermis is a very thin, loose layer which contains many minute bubbles. Then follows a layer with relatively higher density and greater extension.

These three layers altogether correspond to the "skin" which has been found optically.† We cannot analyze the fine structure of the epidermis with the electron microscope. But it is very noteworthy that the dense inner layer which forms the main part of the skin is porous in character (see Figures 5, 6, 7, and 9). Because of the scattering of electrons which passed through the layer, the details are intensely diffused, but nevertheless express picturesquely the network of the skin. The hollow spaces in the net should not be so large as to disturb the *statistically* tangential nature of the net. The shape and orientation of the hollow spaces of the net also should satisfy this condition.‡

The net structure of the skin which was revealed directly by the electron microscopic observations is too fine to be resolved optically. Thus, if we compare the figures obtained with the electron microscope with those obtained optically, we will see that many important details have been resolved. But, it is of interest to notice that the optical methods, especially metachromatic dyeing, application of the polarization microscope, and dark-field illumination, proved useful in presenting the most important features of the structure, although broadly.

The dense layers of the skin are followed by a very loose layer, whose structure, as can be seen from the figures, is like that of a sponge (Figures 5, 8, and 10). This layer corresponds to the core of the optical microscopic figures. The periodic occurrence of the dense and loose layers continues further, but it must be noted that the difference between these layers becomes less distinct in the interior of the fiber. As can be deduced theoretically, electron microscopic figures always show higher contrast in density than optical microscopic figures. Hence, it is possible that the inhomogeneity in the central part of the fiber would not be perceived by the optical microscopic observation.

A radically different structure is seen for xanthate fibers, coagulated by ammonium sulfate solution, and also for the fiber samples which were prepared by coagulation followed by decomposition of the xanthate fibers by sulfuric acid. Figures 12, 13, 14, and

FIG. 4. *Cross section of an unstretched coagulated viscose fiber, observed by dark-field illumination with a Cardioid condenser (750×). The sample was immersed in Canada balsam and observed. The gaseous spaces included in the center are so voluminous that each hollow is interpenetrated, and the balsam, which has the same reflex index as cellulose, occupies all these spaces; hence there is no brightening under dark-field illumination.*

* Employment of higher magnification did not result in any notable improvement of the figures. Refer to the later description.

† The dimension and the structure of these layers on the whole correspond to those of the skin found optically.

‡ Refer to the later discussion.

15 show the cross and longitudinal sections of such fiber samples. Neither the succession of layers nor the fine structure of the tissue is seen for the xanthate fibers. The decomposed xanthate fibers represent, however, very fine porous tissue which can scarcely be resolved by our electron microscope, but the layer succession as described does not occur. Therefore, we may conclude that the mechanism of fiber formation in these cases differs fundamentally from that in the case of coagulation in the usual spinning bath which contains sulfates and sulfuric acid.

4. Discussion

Optical and electron microscopic observations have revealed many important features in the structure of unstretched spun viscose fibers. This structure should not be accidental, but be closely connected with the phenomena which took place at the spinning of viscose. We want first to discuss the structure of fibers which were coagulated in the usual spinning bath containing sulfuric acid and sulfate.

As already stated, no satisfactory and infallible general theories on the cause of the Liesegang phenomena have been presented [9]. However, as far as the occurrence of various layers in our examples is concerned, there seems to be no doubt, if we differentiate the action upon viscose of the salts (sulfates) in the coagulating bath from that of the acid (sulfuric acid). According to our extensive studies on the electrolyte coagulation of cellulose xanthate sol by the action of various salts and acids, the action of the hydrogen ion takes place more slowly by far than that of other cations.* Furthermore, the action of the hydrogen ion is intensely "antagonized" by that of other cations.

Therefore, when viscose is extruded into a spinning bath containing sulfates and sulfuric acid *in proper proportion,* the surface of the extruded viscose is subjected first to the action of the sulfate, forming a very thin, coagulated sheath-like layer, which is an early stage in the formation of the epidermis. The cation of the sulfate is consumed within this layer (owing to the adsorption at coagulation), and the bath liquid loses its original balanced proportion of sulfate to acid. Consequently, the adjacent interior is exposed to the action of the degenerated liquid rich in hydrogen ion, which, now free from the antagonizing action of the sulfate cations, decomposes viscose violently, accompanied by the evolution of gases. The thin, loose layer (the second layer) which lies just underneath the epidermis and contains many minute gas bubbles is thus formed. Hydrogen ion is consumed in its turn in this layer.

In the course of time, the fresh bath liquid penetrates through these layers farther into the interior and the regularly ordered phenomena described above take place here also. In this way the third and fourth layers are formed. The further layer succession can be explained similarly.

Such alternative occurrences of dense and loose layers take place in similar fashion, but not identically. Here we recall that intense dehydration takes place at precipitation of cellulose from cellulose xanthate sol, accompanied by a remarkable decrease in volume of colloidal particles.* At the same time, the water which was set free begins to diffuse out by osmosis into the bath liquor through the semipermeable layers just formed. Thus, intense flow of liquid takes place from the interior to the outer layers, and the outer layers are exposed to the compression caused by the osmotic pressure. The more external the layer the more intensely it is affected by the compression. Hence, the first layer is compressed to the highest compactness, causing at the same time the tangential arrangement of the structural units.† This is the structure of the epidermis.

On account of poor electron transmission, the fine structure of the epidermis could not be revealed by our electron microscopic examinations, but if we notice the shape of gas bubbles in the thin, loose layer just below the epidermis, then we can see how the structure is affected by the compression mentioned above (see Figures 6 and 7). The effect of the compression is seen also in the third layer—that is, the main part of the skin. Careful observation of Figures 6, 7, and 9 suggests that the hollow spaces are shaped as if they were compressed radially. Of interest is the shape of sharply outlined air bubbles, whose cross sections, as can be seen clearly in Figure 5, are not circular, but so elliptical as to indicate the compression described above. This compression causes the tangential net structure of the "skin."

* The actions of salts themselves differ greatly in the ability and speed of precipitating cellulose xanthate sol. For instance, zinc ion has not only high flocculating ability, but also extremely rapid action.

* Dehydration occurs also at the colloid chemical flocculation of the xanthate sol, but in less degree.

† We recall that no drafting has been applied.

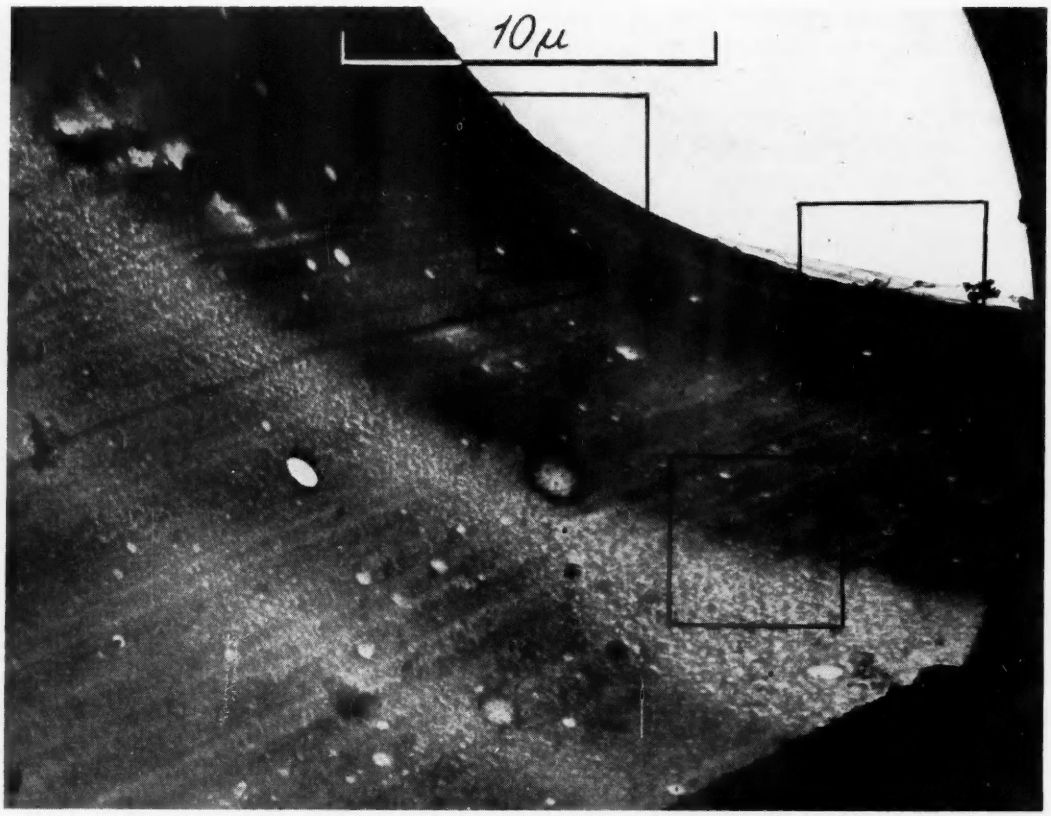

FIG. 5. *Cross section of an unstretched coagulated viscose fiber, observed by electron microscope (1,000 × → 5,000 ×). Viscose: cellulose 8 percent, total alkali 6 percent, ammonium chloride number 10. Coagulating bath: sulfuric acid 10 percent, sodium sulfate 25 percent. (For the succession of layers and features of the structure, refer to the description in the paper.) The sharply outlined bubble-like figures may be tiny air bubbles which were occluded from the first in the viscose.*

On the contrary, the fourth layer is formed by the action of degenerated liquor rich in hydrogen ion. Intense disturbance due to the evolution of gas with the formation of many large gas bubbles opposes the action of compression, which is weaker here. This hinders the building up of a pronounced tangential structure. The feature of the inner loose layers is thus represented.*

As the flow of liquid proceeds from the center outward, and the precipitation of cellulose takes place under this flow, the concentration of cellulose in the interior of the fiber is lessened. This can actually be seen by observing in Figure 5 the density of each layer from the outer to the interior and comparing the densities of corresponding layers. The more diffuse transition from layer to layer and the less distinct difference in the density of layers in

FIG. 6. *The portion in the square at upper left in Fig. 5 enlarged optically (1,000 × → 15,000 ×).*

* Even the second layer is disturbed too intensely to be pronouncedly tangential. Refer to the polarization microscopic examinations, Figures 1 and 2.

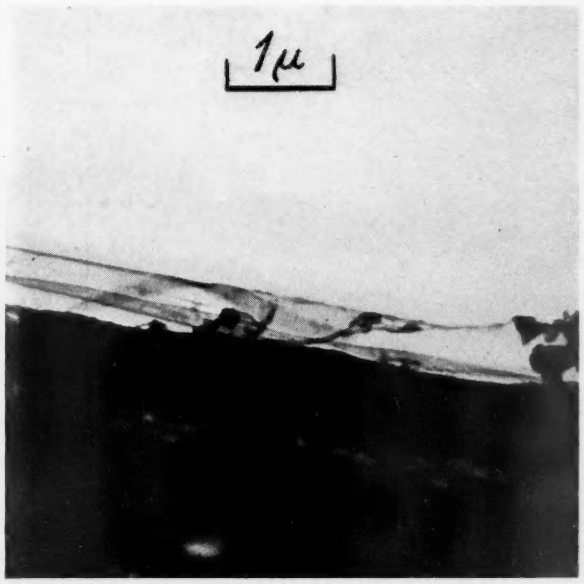

FIG. 7. *The portion in the square at upper right in Fig. 5 enlarged optically (1,000 × → 15,000 ×). The outline of the image is still satisfactorily sharp (see the edge of the image). Resolving power of the electron microscope and photographic emulsion employed is large enough to give sufficiently enlarged detailed figures. The transparent image above the epidermis may be that of a foreign substance (agar-agar?).*

the interior are also easily explained by this fact. The disturbances due to the violent evolution of gas might affect the final structure also.

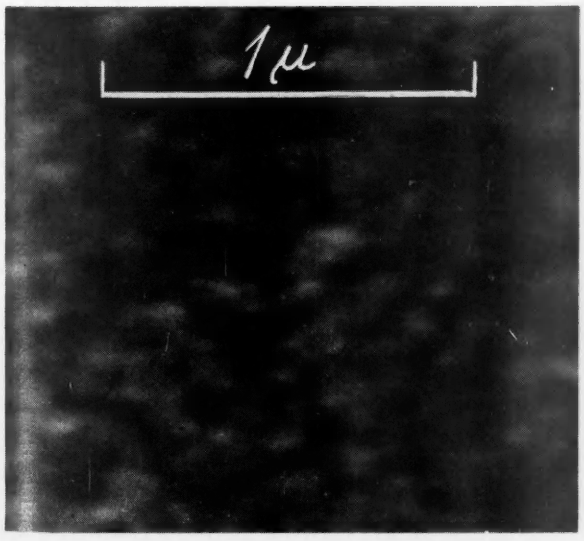

FIG. 9. *The third compact layer (including the epidermis) observed by an electron microscope with a higher magnifying power (9,000 × → 45,000 ×).*

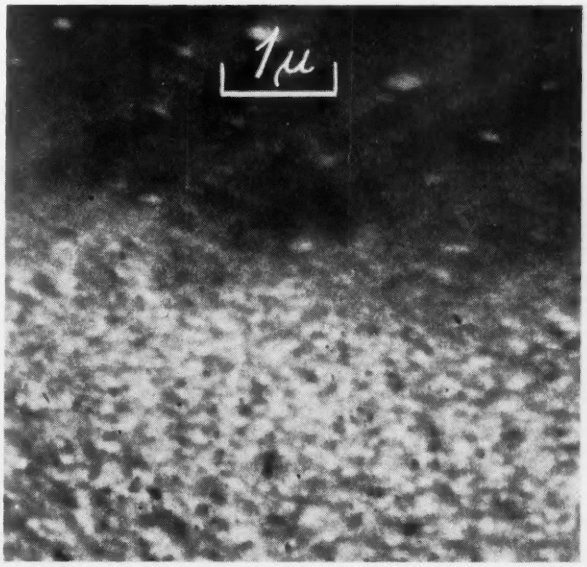

FIG. 8. *The portion in the lower square in Fig. 5 (transition portion from the skin to the "loose" layer) enlarged optically (1,000 × → 15,000 ×).*

The above-mentioned chemical and colloid-chemical phenomena cannot be influenced by mechanical actions such as drawing and stretching. Hence, the skin which has been seen in unstretched spun fibers is formed in the same way as that of fibers which are drawn and stretched in the usual way during spinning. These mechanical conditions cause only attenuation of filaments and transition of the tangential orientation to the axial one. When viscose is drawn and stretched during spinning, the two portions which form the skin and inner layer are elongated, and in the same degree. Therefore, the proportion of the skin is not affected by these mechanical treatments, as was found to be the case. We believe that the conclusion drawn by Preston and further developed by several investigators regarding the cause of skin formation must be revised. According to our interpretation, the freely coagulated fibers can be considered a magnified model of ordinary spun viscose fibers. By the aid of this model we are able to reveal more details of the construction of the viscose fibers and to explain the phenomena which take place at the spinning of viscose.

From the discussion above it is self-evident that the layer succession cannot occur when the viscose is coagulated by a liquid containing neutral salts only. By the action of the salts the viscose is coagulated to form fibers consisting of a network of cellulose

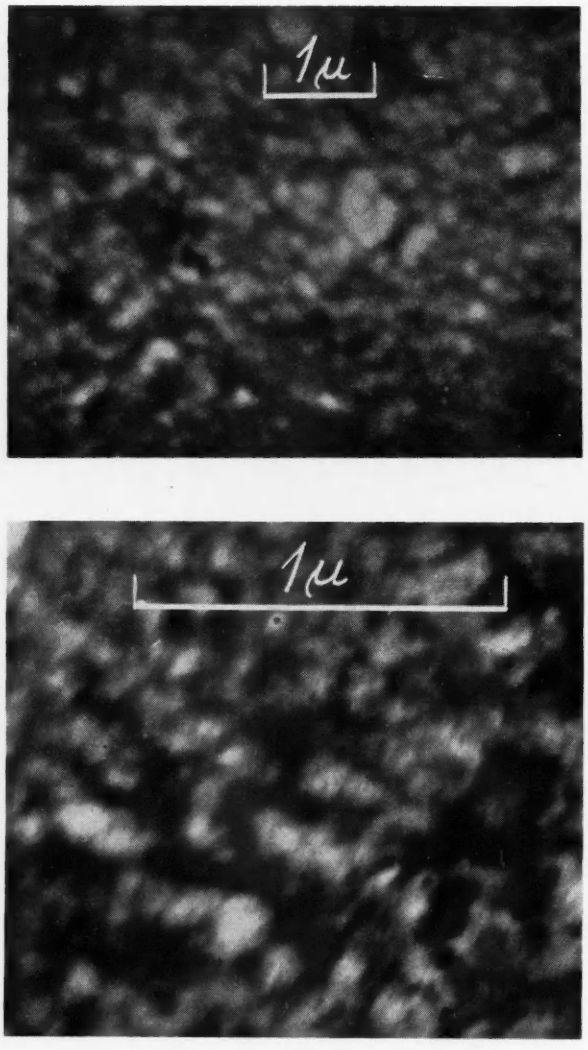

FIG. 10. *Same section as in Fig. 8 (loose layer) observed by an electron microscope with a lower (1,000 × → 15,000 ×) and a higher magnification (9,000 × → 45,000 ×). Higher magnification with the electron microscope did not exhibit any substantial merit in our examination* (cf. *also Fig. 9*). *Higher magnification by electron microscope is more effective only for objects that scatter electrons to a small degree.*

xanthate molecules.* In this case the possibilities for producing such an inhomogeneous tissue as in the previous case are excluded. The coagulation is brought about by the action of salts colloid-chemically, without accompanying evolution of gas and violent dehydration as in the previous case, thus producing a uniform net structure. If we recall that the resolving power of our electron microscope is 5–10 mμ, it follows that the structure of the fibers thus obtained is finer than 50–100 Å.

The structure of isotropic xanthate fiber is indeed notably deformable, as was shown in detail especially by Hermans [16], but the structure in a macromolecular sense is approaching that of the solid state. Hence, the possibility of fundamental interchange of the positions of macromolecules or their swarms is excluded in this structure. Therefore, even if we decompose the xanthate by acid, its original macromolecular structure is not changed radically. This results in forming a homogeneous net structure even after cellulose is regenerated from xanthate fibers.

When xanthate fibers are subjected to the action of sulfuric acid, evolution of gases takes place likewise, but to a lesser extent than in usual spinning, because most of the by-products capable of evolving gases have been removed beforehand. Evolution of gases exerts action to enlarge the hollow spaces of the net, forcing cellulose molecules aside. On the other hand, the removal of xanthate groups makes the coherent power of hydroxyl groups of cellulose molecules more effective, and deformable portions of cellulose molecules are attracted to each other. The pressure of evolving gases assists this attraction, and gas bubbles are occluded in the net in such a manner that the gas pressure keeps equilibrium with the confronting force of the cellulose net. As a result, the tissue becomes less homogeneous, and the porous tissue can be recognized with the electron microscope (Figure 14).

But, if we recall that the xanthate fiber is in a macromolecularly solid state and that the xanthate molecules are fixed at many isolated points which act in hinge-like fashion, then the regeneration of cellulose from this tissue cannot disturb the original structure fundamentally. The portions which lie between jointed points of molecules can be deformed or forced aside and attracted together closely. There-

* In what state cellulose xanthate is dissolved in the usual viscose is a very important point—whether as colloid particles, the crystallites of alkali cellulose (Na-Cell I), as was assumed by Hess [10], Schramek [11], Lieser [12] etc., or macromolecules of cellulose xanthate, as was concluded by Staudinger and coworkers [13], and Bredee [14].

The solution state of viscose is now a subject of study, on which we are concentrating our interest. According to the result of our recent study on the diffusion of cellulose xanthate we can presume that the particles are not pre-existing micelles of alkali cellulose, but appear to be swarms of chain molecules crumpled together. Hermans [15] assumed also similar configuration from the deformability of isotropic cellulose fibers.

FIG. 11. *Cross-section sample of fiber similar to that of Fig. 5 was prepared by sectioning the wedge (1,000 × → 7,500 ×). Stripes which run parallel in one direction are due to unevenness of the knife edge. The section was supported on celloidin foil, which can be seen in the photograph. Even an extremely slight difference in thickness of layer brings about notable differences in transmissibility of electrons. The gradient of thickness in the wedge is not so large as we might expect it to be from this photograph and Fig. 12.*

FIG. 12. *Cross section of a cellulose xanthate fiber (1,000 × → 5,000 ×). Viscose was extruded from a spinneret into a saturated solution of ammonium sulfate and sectioned by the wedge-section method. Fine structure, as in Figs. 5–11, cannot be seen within the limits of the resolving power of our electron microscope* (cf. *also next photographs*). *Images of air bubbles occluded in the viscose are outlined sharply at the extremity (thinner layer) of the wedge, whereas those in the thicker layer (scattering of electrons) are diffused.*

fore, the fineness of the net of regenerated cellulose fibers from xanthate cannot be accidental, but is determined *a priori*. From Figure 14 we may estimate the fineness of the tissue at about 1,000 Å.

It is obvious that the intense flow of liquid due to osmosis cannot occur at the decomposition of xanthate fibers by sulfuric acid, and the structure is free from the effect of compression. If we observe the structure shown in Figure 14 carefully, we will see that the net is practically isotropic. The shape of sections of air bubbles also is circular, without showing any indications of the effect of compression.

It is very difficult to compare the density of the tissue of the different samples accurately, because we are not able always to prepare cross-section samples of the same thickness. But, according to our impression of electron microscopic observations, it is certain that the solid part in the skin of fibers prepared by employing an ordinary bath is constructed with cellulose chains which are compacted more closely than in the tissue of fibers prepared by regenerating cellulose from xanthate fibers with acid. The net structure of the skin, in spite of the fact that it contains larger hollow spaces due to gases, might have higher mechanical strength (per unit cross-sectional area) than that of regenerated cellulose fibers from xanthate. Furthermore, the weight percentage of cellulose in the skin is larger by far than

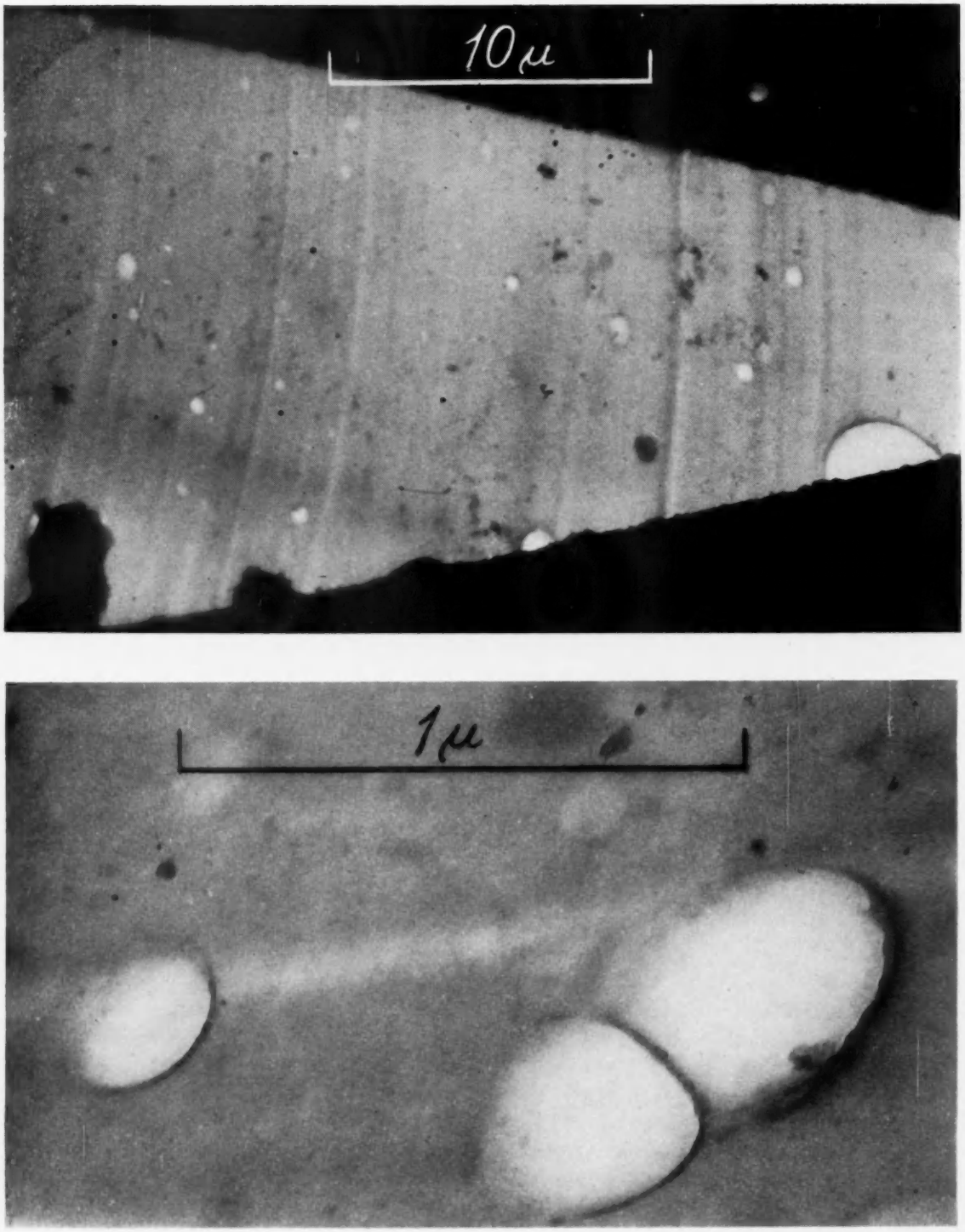

FIG. 13.

A (top)—A fiber sample similar to that in Fig. 12 was sectioned by the usual microtome method (1,000 × → 7,500 ×). Dark shade shows the mesh wires. The edge of the wires is diffuse at the background of the tissue, whereas it is sharp under a bubble. This also is due to the scattering of electrons by the tissue.

B (bottom)—A similar sample was observed at a higher magnification (9,000 × → 90,000 ×). In the portions containing bubbles the thickness of the layer would be sufficiently small to be free from the effect of scattering, and yet we cannot recognize any fine structure.

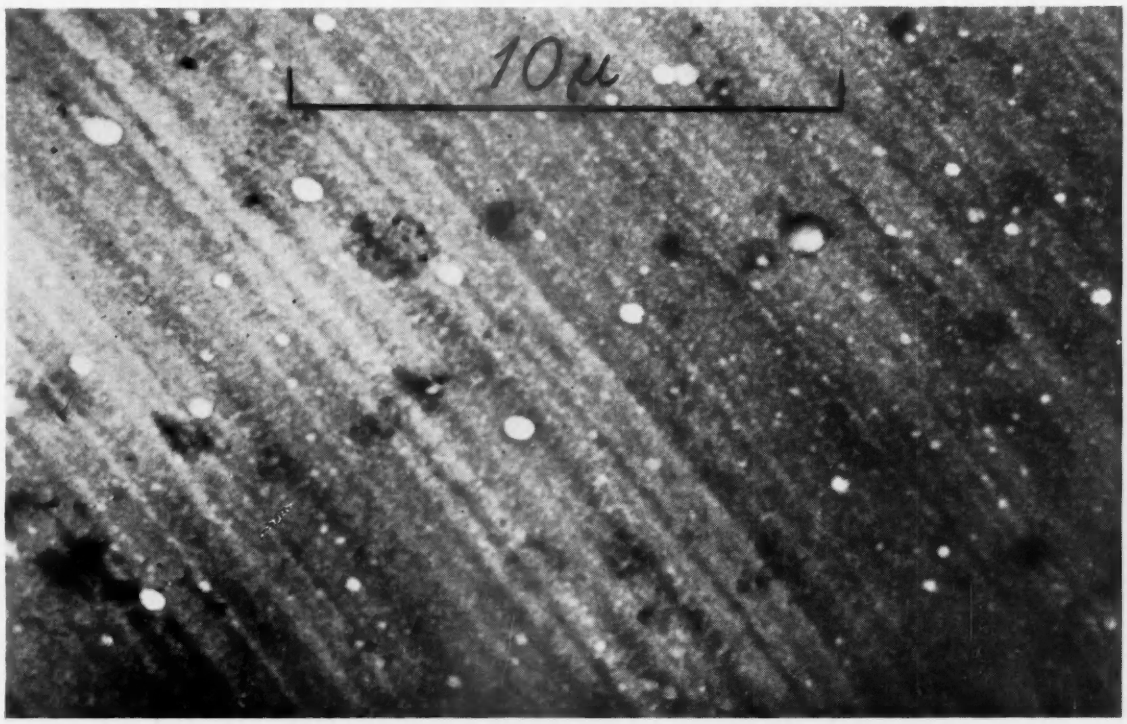

Fig. 14. *Longitudinal section of a regenerated cellulose fiber from coagulated xanthate fiber (1,000 × → 7,500 ×). Very fine structure of an isotropic nature is seen. Shape of the sections of air bubbles is almost circular.*

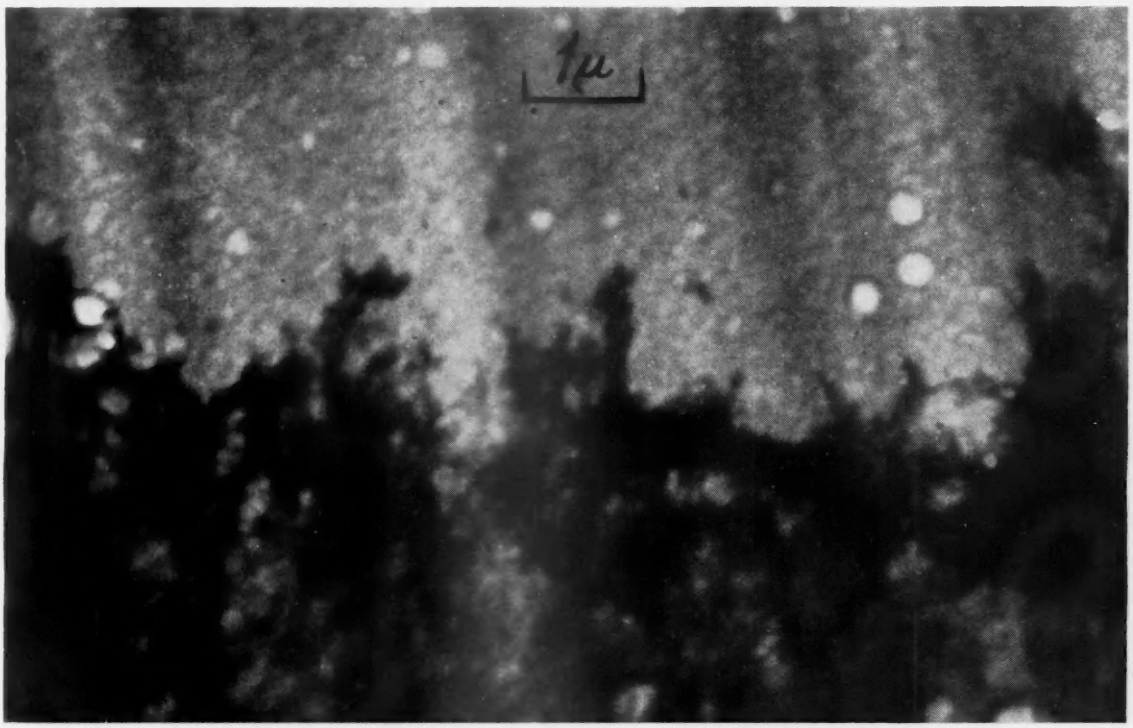

Fig. 15. *Similar photograph to Fig. 14 (1,000 × → 15,000 ×). Darker figure shows the burnt residue of the former photographing. We can distinguish such failure figures from the normal ones with certainty.*

that of cellulose in the loose inner layer.* This suggests that stronger cellulose fibers can be obtained by the use of a well-balanced ordinary bath, containing sulfuric acid and sulfates, than the cellulose fibers made from stretched xanthate fibers. We believe that this has been actually justified.

Compact bundles of cellulose chains result from the crystallizing action due to the molecular cohesion of hydroxyl groups. When the usual spinning bath is employed, the cellulose xanthate molecules which are dehydrated in appropriate degree by the action of the sulfate cations are still capable of being translated when subjected to the action of the acid. As the xanthate groups are removed, the hydroxyl groups, instead of xanthate groups, become available for crystallization, and the chains exhibit a stronger molecular cohesion. Could stretch be applied promptly just at this stage, then axial orientation and its fixation would be brought about successfully.

On the contrary, the chains of cellulose xanthate are less favorable for crystallization, because of stronger hydrating property and larger spatial occupation and hindrance of xanthate groups.

In order to effect a higher degree of crystallization, fibers must be stretched just when hydroxyl groups are being set free by the action of the acid.† It is an important problem to study how the structure as shown by the electron microscope would be changed when we draw and stretch the viscose during spinning. At the same time, it is of interest to study the submicroscopic structure of fibers which are made from the low caustic viscose, which contains remarkably smaller amounts of the by-products that evolve gases during spinning.‡

5. Experimental Data

A. Electron Microscope

An electron microscope with two electrostatic lenses was employed.§ To eliminate the influence of spherical aberration the pinhole diameter of the objective was made as fine as 0.1–0.05 mm. at the sacrifice of brightness of images. Harmful discharge between lens electrodes was avoided by carefully finishing the surface to the greatest possible smoothness. For ease and good performance of working and finishing of lenses the middle electrodes were made of special stainless steel, while the outer electrodes were made of chromium-coated bronze. By means of microscopic examination, scrupulous care was given to the least roughening of the surface. Nevertheless, it seemed that a heavier loading than 40 kv. was not allowable.

Evacuation up to 10^{-5} mm. Hg was attained by employing a diffusion pump of the Leybold type, combined with a rotary pump and provided with a trap, filled with active carbon.

The magnifying power of the objective was 100, that of the projecting lens 10, and the total magnification was, accordingly, 1,000 times. In order to obtain higher magnification an objective of 90 times and a projecting lens of 100 times (the total magnification 9,000 times) were employed. However, such higher magnification did not prove highly efficient for our samples (see Figures 9 and 10).

Each magnified image is made visible by means of fluorescent plates, with which the apparatus is furnished.

The resolving power was estimated at 5–10 mμ.

At this point mention should be made of the reason for adopting lower magnification. As will be shown later, the special techniques are needed for the preparation of the objects of fibrous materials for electron microscopic investigations. Notwithstanding, it may be said that a satisfactorily thin object is not always obtainable. On account of the scattering of electrons, 5 mμ is the limit of the resolving power of the electron microscope when organic substances are the objects of study. On the other hand, the limit of the distinguishing ability of the human eye is estimated at 0.1 mm. Therefore, the under limit of the effective magnification is 20,000 times. Higher magnification than this is of service only in aiding the eye to sight the object and does not heighten the sharpness of the image.

The resolving power of the fine-grain photographic emulsion reaches 5 μ. This means that optical magnification as much as 20 times is useful for improving the image (compare Figure 5 with Figures 6, 7, and 8). For this reason, an instrument with a magnifying power of 1,000 times is identical in its

* The area percentage of the skin in the cross sections of well-formed fibers of 1.5 denier exceeds 70 percent (stretch-spinning with the two-bath process).

† Occurrence of the optimum iodine number at the stretch point, described in detail in another paper.

‡ It has been shown that the amount of by-products carbon bisulfide of the low caustic viscose is always remarkably smaller than that of usual viscose, while the amount of combined carbon bisulfide does not differ perceptibly.

§ The construction was based principally upon the design of Mahl. The original construction of the Tokyo-Shibaura Electric Works was improved by us in several respects. We are indebted to Dr. S. Asao for the construction of the electron microscope and for his kind advice.

ability with an instrument with a magnifying power of 20,000 times, providing the resolving powers of both are identically 5 mμ.

There are, in addition, two other merits of lower magnification. The current density of electrons which gives the same intensity of illumination on a fluorescent screen is inversely proportional to the square of the magnification. Hence, we can do with by far smaller current density and can be free from the danger of burning and melting the samples. Results of several investigators such as Ruska, Jentgen, Zahn [8], and others who adopted higher magnification lose their value.

The magnifying power of 1,000 times is exactly the limit we are able to reach optically. Therefore, if we employ an electron microscope with this magnifying power, it is easy to compare the images of the electron microscope with those of the optical. Consequently, we can discuss the figures obtained with the electron microscope with great confidence, knowing "what portion of the specimen" was observed "from what direction." And there is no danger of faulty interpretations as there is when the examinations are made with the electron microscope alone.

B. Preparation of Sectioned Samples

In order that a satisfactorily detailed image may be obtained with transmitted electrons the object layer must be extraordinarily thin.* We investigated two methods of sectioning—that is, (*a*) the method of wedge-shaped section and (*b*) the usual microtome method. Both methods proved of use for our samples. But special techniques and precautions were needed for embedding and sectioning.†

1. Embedding. When sections of fibrous specimens are prepared in the usual way, air-dried fibers or fibers which have been dehydrated with alcohol, toluene, etc., are embedded in candle paraffin and sectioned by a microtome.

Closer adhesion between fibers and paraffin was necessitated in order to obtain thinner slices. It was found that the usual method of embedding is unsatisfactory for this purpose, for the fibers are likely to be displaced in the direction of the stroke of the microtome knife and the sections of fiber may come out of the paraffin slice. To avoid this, we tried embedding the fibers in agar-agar first, and then in paraffin. This has produced very satisfactory results.

The fibers stored in water were taken out of the vessel, freed from the excessive water by wiping with filter paper, and placed in parallel arrangement in hot agar-agar solution.* The favorable concentration of the agar-agar solution was about 5 percent. After the agar-agar was cooled and solidified, a small rectangular block was cut out lengthwise the fibers.

Prior to embedding the agar-agar block in paraffin, all the moisture must be removed from the agar-agar column and the specimen. The stages of the process were as follows: The block was immersed in 50-percent, 75-percent, and 95-percent ethyl alcohol each for 30 minutes and in absolute alcohol 2 hours. After the moisture was completely removed, the block was soaked in alcohol-xylene mixture (1 : 1) for 2 hours and in xylene for 2 hours. The block was then immersed in xylene-paraffin mixture for 5 hours at 70–75°C and in melted paraffin for 5 hours at 70–75°C in a thermostat.† It is important that paraffin penetrate thoroughly into the tissue of both the agar-agar and the specimens. Finally, we cut out a paraffin block which contained the agar-agar block.

It was found that the combination of paraffin and agar-agar tissue was just what we desired, if we did not heat the agar-agar too long in the paraffin.‡

* According to Ardenne [17], the object layer must be as thin as 10^{-4} mm. (0.1 μ), in order to reach the resolving power of 10^{-3} mm. (10 μ), provided organic substances are the objects and the electron speed is 50 kv. Judging from the sharpness of our figures in this paper we can conclude that the desired thickness was attained.

† It might be expected that satisfactory preparations for electron microscopic observations could not be obtained from the usual viscose filaments which have been spun-stretched. Actually, we met with difficulty in obtaining sections of these filaments, the skin, where the structural units are strongly oriented and make up too compact tissue, exerting higher resistance to the movement of the microtome knife and being liable to cleave at the extremity of the wedge. In order to advance the present study, we must obviate this difficulty and it is our intention to accomplish this.

* Agar-agar is dissolved in boiling water, and the solution is allowed to cool at room temperature. The solution becomes viscous little by little. When suitable viscosity was reached a mass of agar-agar was heaped up on a glass slide and then we embedded a bundle of fibers in it.

† The temperature at which the paraffin is kept in the molten state is favorable. Too-high temperature is likely to cause excessive hardening of the agar-agar tissue.

‡ Employment of celloidin instead of agar-agar did not produce satisfactory results. Celloidin became so hard during the soaking in paraffin that the knife edge of the microtome bent slightly and vibrated slightly when it cut off the sections, and consequently the surface was slightly waved.

It was very effective to admix about 20 percent of Japan-wax * with the paraffin to reduce its crystalline character and to increase the toughness.

The melting point of paraffin should be 65°C at least. It is recommended that before use the paraffin be heated long enough for partial oxidation to take place, in order to reduce the size of crystalline particles.

Removing paraffin and agar-agar from the sections was very easy if we dissolved out the paraffin with xylene, drove it out with alcohol, and added water and then warmed them. These procedures can be performed easily on a slide glass if care is exercised.

2. Sectioning. (*a*) The Method of Wedge-Shaped Section: This method was recommended especially by Ardenne [18], who has constructed a special arrangement for inclining the object desk. However, since we employed a sliding microtome, no special inclining apparatus was needed, but a Neapler sample-holder, which is a well-known accessory of microtomes, was used.

Selection of a good knife seemed to be of essential importance. We employed a knife of Walb, the upper surface being concave and the other plane. Before use, the knife was polished and finished thoroughly, and the knife edge was examined with a microscope (200 and 600 ×).

The knife was attached to the microtome at an angle of about 45° to the direction of its stroke. The upper layer of the sample was cut off to expose the surface containing the specimens. When the Neapler holder was screwed the surface was tilted 2–3°, the side against which the knife edge strikes being higher than the other side. Here, the surface to be sectioned was coated with melted paraffin and allowed to cool. It is necessary to coat the surface with a fresh paraffin layer, in order to prevent the deformation of the section at its wedge extremity. Then a section was sliced off. Position of the sample must be so adjusted that the extremity of sliced wedge contains the cross sections of the fibers.

Use of the same portion of the knife edge for more than one sectioning was avoided. After each sectioning the knife was displaced and fixed so that another marked portion became available for the next sectioning.†

Every several slices were placed in a glass tube filled with xylene, and the paraffin was dissolved out, the tube being shaken very slightly if necessary. Deparaffinized sections were washed with alcohol and stored in water. Alcohol with the strength of 75 percent proved to be most favorable, as the sections became somewhat opaque in this liquid and could be distinguished easily.

(*b*) The Usual Microtome Method: The usefulness of this method was determined experimentally.* Both sliding and rotary microtomes were useful, providing the step of the feed screw is smaller than 1 μ. We employed both a rotary and a sliding microtome, whose step of screw is designed to be 1 μ (rotary) and 0.5 μ (sliding) respectively.

With the step which advances the sample set at the minimum, successive sectionings were performed. The cut sections form a "ribbon." Microscopic examination of the sections in such ribbons reveals that usually they are not identical in thickness, owing to the bending of the blade edge at sectioning and lack of precision of the screw. Thinner sections could be picked up out of several ribbons.

C. Mounting the Section

Tungsten and tantalum screens of 200 mesh were used for holders. To prevent possible disturbances due to the secondary emission the round wires were flattened by pressing the meshes with a roller press. The mesh was hollowed, basin-like, and the specimens were mounted in the center of bottom. The specimens examined in this study were sufficiently large to cover a hole of the mesh. Employment of the celluloid or celloidin foil as an auxiliary supporter did more harm than good.

Summary

1. Cross and longitudinal sections of freely coagulated viscose fibers were examined by optical and electron microscope.

2. The electron microscopic observations revealed many submicroscopic features of the structure which have not been disclosed optically.

3. The "skin" is formed regardless of drafting or stretching during spinning. The opinion offered and developed by Preston and several other investigators must be revised.

* Other substances which have a similar effect may be substituted.

† In spite of these precautions the sections contained many stripes which are caused by unevenness of the knife edge, as can be seen in Figures 11 and 12.

* Figures 5–10 and 13–15 were obtained by this method. If we compare these photographs with Figures 11 and 12, which were obtained by the wedge method, we notice that this method produces rather better results.

4. The coagulation of viscose by the usual coagulating bath is a typical example of Liesegang effect.

5. The submicroscopic structure revealed especially by the electron microscope was correlated with the phenomena which take place at the spinning of viscose.

The cause of skin-formation, and, more generally, the cause of alternative occurrence of layers were explained.

6. Xanthate fibers and cellulose fibers from xanthate show utterly different microscopic and submicroscopic structure. In these cases fibers are formed in radically different ways.

7. Submicroscopic structure revealed by the electron microscope can be correlated most closely with the mechanical properties of the fibers.

Acknowledgment

The authors desire to express their grateful acknowledgment to Hon. Prof. G. Kita for his kind encouragement, which accelerated our studies. They would also like to express their indebtedness for the financial support of the Research Institute of Artificial Fibers, Japan (Nippon Kagaku Sen'i Kenkyusho), Taniguchi Foundation for Promoting Industry (Taniguchi Kogyo Shoreikai), and the Society for Advancement of National Arts and Science (Nippon Gakujitsu Shinkokai).

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   (*b*) Ruska, H., *Kolloid-Z.* **92**, 276 (1940). (Cellulose fibers.)
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   Conditions of samples discussed in the above papers: (*a*) unknown; (*b*) powdered; (*e*) swelled and pressed; (*d*), (*f*) ground; (*c*), (*g*), (*j*) chemically treated; (*h*), (*i*) sectioned (but imperfectly).
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Correction

In the paper "Geometrical Principles Applicable to the Design of Functional Fabrics," by Dr. F. T. Peirce, published in the March, 1947, issue, there is an error on Figs. 1A and 1B on page 138.

The quantity "D" shown on both figures should be "D/2." D represents the sum of the diameters of warp and filling; the distance between axes of warp and filling is half this quantity.

Laboratory Techniques

The suggestion has been made that there be included in TEXTILE RESEARCH JOURNAL *a special section for notes on laboratory techniques peculiar to textile testing and research which would ordinarily not be of sufficient importance or length for publication as a formal* JOURNAL *paper.*

Such a section would invite free exchange of helpful modifications of existing tests or apparatus for special purposes and bring to light newly developed methods and devices. The time spent in standardizing present tests and and developing new ones for the multitude of problems facing the textile technologist would be considerably reduced if information of this sort were made available. Therefore the JOURNAL *invites contributions from its readers.*

In accordance with the established practice, all papers will be submitted to competent judges before being accepted for publication.

The paper by Seaman J. Tanenhaus, below, falls within the scope of the plan.

The Median as a Typical Value for Walker Yarn Abrader Data

Seaman J. Tanenhaus*

Abstract

The data obtained on the Walker yarn abrader in the determination of abrasion-resistance of yarns form frequency distributions which are decidedly positively skewed. The mean of such distributions, being affected by the extremes, tends to be atypical. The modal class is too broad for precise differentiation and the first breaks are too variable to be of use. The median, which is independent of the extreme values, is more representative. Furthermore, by computing the average median of several sets of yarns rather than determining the lot median, a saving of time of approximately 50 percent is effected.

A phase of the research program on the "Wear-Resistance of Textiles" entails the critical evaluation of laboratory apparatus and procedures in an effort to ascertain the significance of test results. A cursory examination of the data obtained on the Walker yarn abrader, an instrument for determining the abrasion-resistance of yarns, indicated that the results form frequency distributions which are decidedly skewed to the right. The non-normality of the data distribution indicates that those statistics usually considered sufficient—that is, the mean, standard deviation and number of observations [1]—may be inadequate in this case. As it is important that the chosen value represent the level of the characteristic being investigated, a study was made of the possibility of substituting some other criterion for the generally accepted mean.

* Technologist, Textile Research Laboratories, Philadelphia Quartermaster Depot.

Apparatus and Material

The Walker yarn abrader is an instrument by which yarns under a given tension are rubbed against themselves so as to produce a controlled amount of localized wear. The number of cycles producing rupture of the yarn is taken as the end-point for that yarn. Twenty-four individual yarns, which constitute a set, are tested simultaneously.

For this study, the data representing the abrasion-resistance of six lots of yarns subjected to various finishing treatments were chosen at random. Each of the six lots contained ten sets of twenty-four yarns. The data presented in Table I were obtained

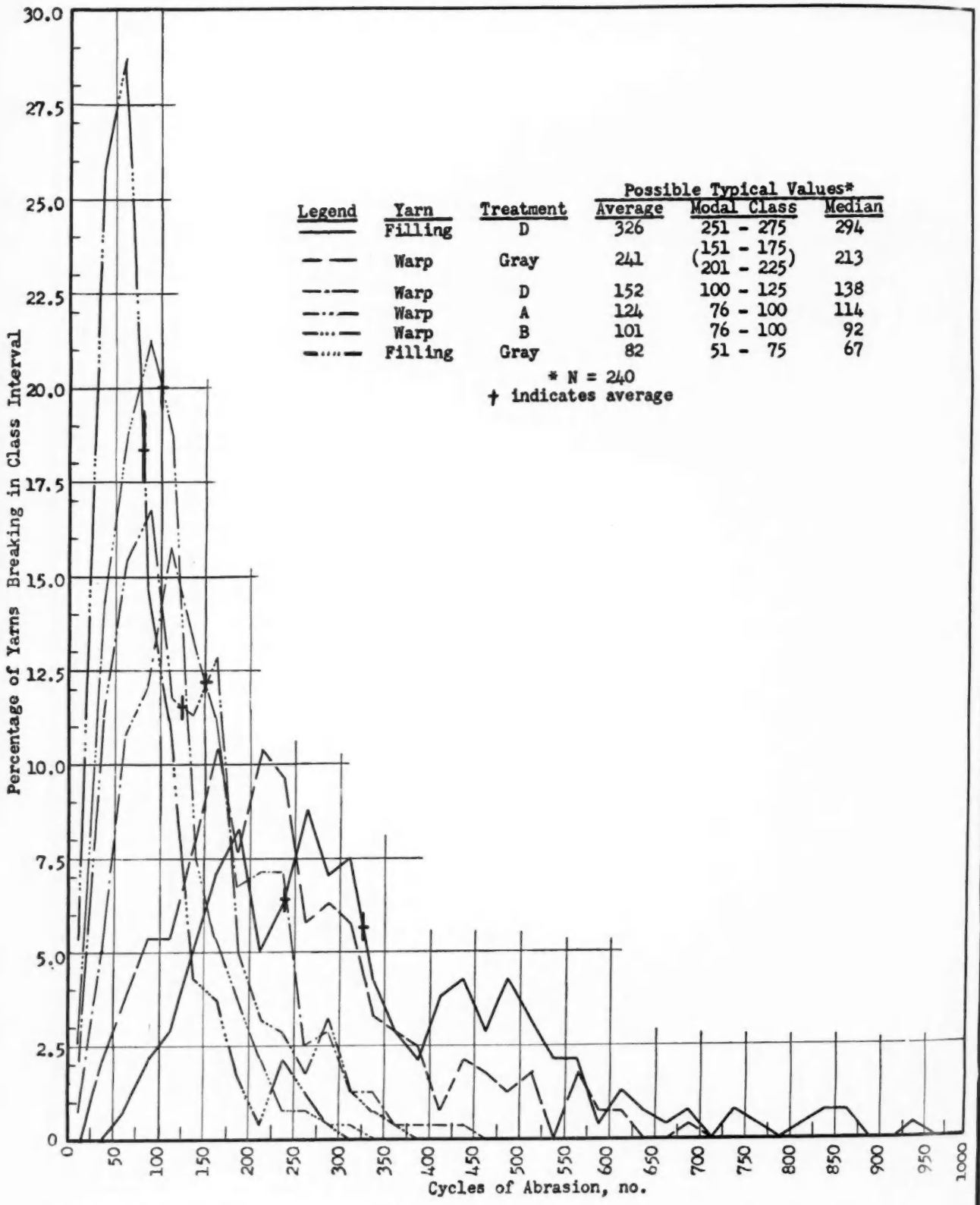
Percentage of Yarns Breaking in Class Interval
30.0
27.5
25.0
22.5
20.0
17.5
15.0
12.5
10.0
7.5
5.0
2.5
0
0 50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000
Cycles of Abrasion, no.
Legend | Yarn | Treatment | Possible Typical Values* Average | Modal Class | Median
Filling | D | 326 | 251 - 275 | 294
Warp | Gray | 241 | (151 - 175, 201 - 225) | 213
Warp | D | 152 | 100 - 125 | 138
Warp | A | 124 | 76 - 100 | 114
Warp | B | 101 | 76 - 100 | 92
Filling | Gray | 82 | 51 - 75 | 67
* N = 240
† indicates average

FIG. 1. *Frequency distribution of abrasion-resistance of six groups of yarns (Walker yarn abrader). Data from Table I.*

on an instrument known to be in statistical control by the utilization of control-chart techniques as recommended by the American Society for Testing Materials, a 2-ply combed peeler mercerized yarn having been used as the check material. The test yarns themselves were ascertained to be in control by the same technique, but the data were rearranged into frequency distributions for the purpose of this work.

Procedure

The data were first plotted in the form of frequency distributions, as shown in Figure 1. The curves illustrate graphically the skewness of the distributions of the number of cycles required to produce yarn failure. The data then were plotted as cumulative frequency distributions on logarithmic-probability paper as a convenient test of logarithmic normality [2]. The resulting straight lines between the first and third quartiles indicate logarithmic normality in this region. As a further check, the logarithmic coefficient of skewness was calculated following the suggestion of Davies and Crowder [3], who state that: "If this coefficient is less than 0.15 or perhaps even 0.20, the distribution may tentatively be regarded as of the logarithmic type." The coefficient is computed from the quartile values by the following formula:

$$Sk = (\log Q_1 + \log Q_3 - 2 \log Q_2)/(\log Q_3 - \log Q_1).$$

For each of the six lots, the value of Sk is less than 0.15.

Discussion

The primary requisite of a representative statistic is that it express the quality of the material under consideration. An additional desideratum is that the desired value be obtained with a minimum of work. To obtain the average abrasion-resistance of a set of yarns, it is necessary to continue a run until all of the yarns fail. However, the validity of the data obtained from those relatively few yarns which appear to be abnormally resistant to wear is questionable, for the durability may not be an inherent characteristic but, rather, a result of the presence of a bearing mechanism formed by the balling-up of loose fibers. Furthermore, these few individual yarns which require an unduly long time to rupture necessitate use of the apparatus and attention of the operator for disproportionate periods. To ascertain whether some other statistic unaffected by the extremes might be used with increased confidence and, if possible, a concomitant saving in time in lieu of the mean, consideration was given to the mode (or modal class), the first break, and the median as possible alternatives. Table II presents the values of the various statistics for the six lots.

In general, each of the various statistics ranks the lots in the same manner. However, the elimination of all except the median is suggested by the following considerations:

The shape of the curves below the first quartile in Figure 2 indicates the probability of non-normal logarithmic distribution of early ruptures, casting doubt upon the advisability of the first break as a criterion.

Although the mode is by definition the most typical value, modal classes are too broad to permit precise differentiation of lots, a condition emphasized by the presence of bimodal distributions (Lot 2, Figure 1, for example). Furthermore, practically all of the individual yarns in a set must be broken before the modal class becomes apparent, thus minimizing the saving in time. The range of the data is so wide, because of the inherent variability of textile components, that the use of modal classes is necessary for intelligent consideration of the data.

As the cumulative frequency distribution curves (Figure 2) are straight lines between the first and third quartiles, thus indicating validity of comparison within these limits, the median is suggested as the most suitable statistic for representing the data. If instead of determining the lot median, the average of the medians of several sets is calculated, a saving in time of more than 50 percent is effected.

Calculating the average median of the individual sets rather than obtaining the median of the entire lot results in a slight error, which is considered to be not significant. The differences between the value of the lot medians and the average medians of the sets are given in Table III.

Conclusions

The best typical value representing quality levels of samples tested on the Walker yarn abrader is the

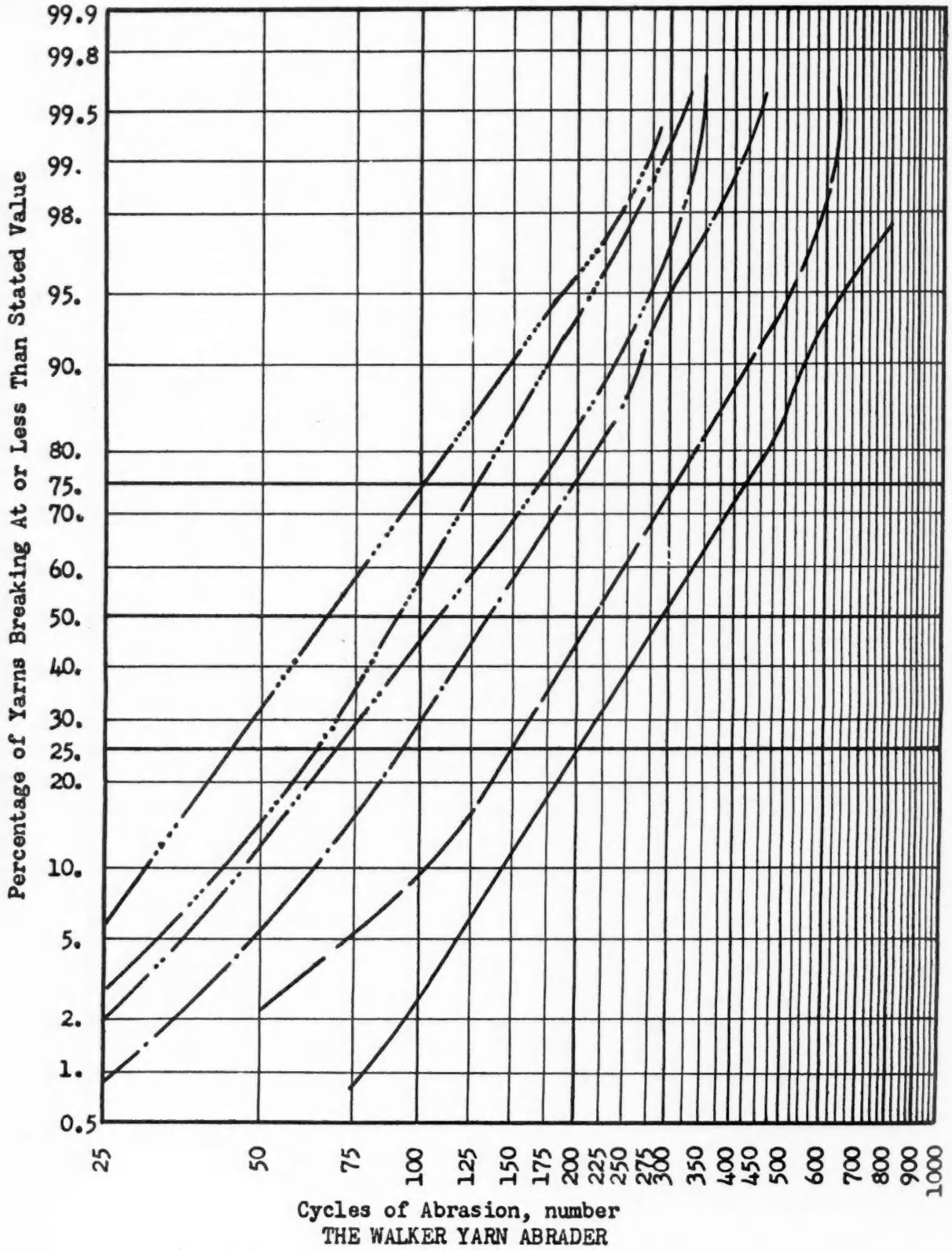

FIG. 2. *Cumulative distribution of abrasion-resistance of six groups of yarns (Walker yarn abrader). Data from Table I.*

TABLE I. FREQUENCY DISTRIBUTION OF ABRASION-RESISTANCE OF SIX GROUPS OF YARNS (AS DETERMINED ON THE WALKER YARN ABRADER)

	Lot 1			Lot 2			Lot 3			Lot 4			Lot 5			Lot 6		
Class	*f*	%	Σ %	*f*	%	Σ %	*f*	%	Σ %	*f*	%	Σ %	*f*	%	Σ %	*f*	%	Σ %
1–25	0	0.0	0.0	0	0.0	0.0	2	0.8	0.8	5	2.1	2.1	7	2.9	2.9	13	5.4	5.4
26–50	0	0.0	0.0	5	2.1	2.1	12	5.0	5.8	27	11.3	13.4	34	14.2	17.1	62	25.8	31.2
51–75	2	0.8	0.8	9	3.8	5.9	26	10.8	16.6	37	15.4	28.8	45	18.8	35.9	69	28.8	60.0
76–100	5	2.1	2.9	13	5.4	11.3	29	12.1	28.7	40	16.7	45.5	51	21.3	57.2	35	14.6	74.6
101–125	7	2.9	5.8	13	5.4	16.7	38	15.8	44.5	28	11.7	57.2	45	18.8	76.0	27	11.3	85.9
126–150	12	5.0	10.8	19	7.9	24.6	32	13.3	57.8	27	11.3	68.5	19	7.9	83.9	10	4.2	90.1
151–175	17	7.1	17.9	25	10.4	35.0	27	11.3	69.1	31	12.9	81.4	13	5.4	89.3	9	3.8	93.9
176–200	20	8.3	26.2	17	7.1	42.1	16	6.7	75.8	12	5.0	86.4	14	5.8	95.1	4	1.7	95.6
201–225	12	5.0	31.2	25	10.4	52.5	17	7.6	82.9	8	3.3	89.7	5	2.1	97.2	1	0.4	96.0
226–250	15	6.3	37.5	23	9.6	62.1	17	7.1	90.0	7	2.9	92.6	2	0.8	98.0	5	2.1	98.1
251–275	21	8.8	46.3	14	5.8	67.9	6	2.5	92.5	4	1.7	94.3	2	0.8	98.8	3	1.3	99.4
276–300	17	7.1	53.4	15	6.3	74.2	7	2.9	95.4	8	3.3	97.6	1	0.4	99.2	1	0.4	99.8
301–325	18	7.5	60.9	14	5.8	80.0	3	1.3	96.7	3	1.3	98.9	1	0.4	99.6	0	0.0	99.8
326–350	10	4.2	65.1	8	3.3	83.3	3	1.3	98.0	2	0.8	99.7	0	0.0	99.6	0	0.0	99.8
351–375	7	2.9	68.0	7	2.9	86.2	1	0.4	98.4	1	0.4	100.1	1	0.4	100.0	1	0.4	100.2
376–400	5	2.1	70.1	6	2.5	88.7	1	0.4	98.8									
401–425	9	3.8	73.9	2	0.8	89.5	1	0.4	99.2									
426–450	10	4.2	78.1	5	2.1	91.6	1	0.4	99.6									
451–475	7	2.9	81.0	4	1.7	93.3	0	0.0	99.6									
476–500	10	4.2	85.2	3	1.3	94.6	0	0.0	99.6									
501–525	8	3.3	88.5	4	1.7	96.3	0	0.0	99.6									
526–550	5	2.1	90.6	0	0.0	96.3	0	0.0	99.6									
551–575	5	2.1	92.7	4	1.7	98.0	0	0.0	99.6									
576–600	1	0.4	93.1	2	0.8	98.8	0	0.0	99.6									
601–625	3	1.3	94.4	2	0.8	99.6	0	0.0	99.6									
626–650	2	0.8	95.2	0	0.0	99.6	1	0.4	100.0									
651–675	1	0.4	95.6	0	0.0	99.6												
676–700	2	0.8	96.4	1	0.4	100.0												
701–725	0	0.0	96.4															
726–750	2	0.8	97.2															
751–775	1	0.4	97.6															
776–800	0	0.0	97.6															
801–825	1	0.4	98.0															
826–850	2	0.8	98.8															
851–875	2	0.8	99.6															
876–900	0	0.0	99.6															
901–925	0	0.0	99.6															
926–950	1	0.4	100.0															
N	240			240			240			240			240			240		

TABLE II.

Lot	Yarn	Treatment	Mean	Possible typical statistics: 1st break*	Modal class	Median
1	Filling	D	326	115	251–275	294
2	Warp	Gray	241	69	151–175 / 201–225	213
3	Warp	D	152	42	100–125	138
4	Warp	A	124	28	76–100	114
5	Warp	B	101	27	76–100	92
6	Filling	Gray	82	20	51– 76	67

* Average of ten sets.

TABLE III.

Lot	Lot median	Average median	Deviation (%)
1	289	294	1.7
2	220	213	−3.2
3	136	138	1.4
4	111	114	2.6
5	93	92	−1.1
6	67	67	0.0
			1.4
		Average deviation	0.23

average median of a number of sets of yarns selected at random.

Acknowledgment

Thanks are expressed to Dr. Lewis Larrick, Institute of Textile Technology, whose suggestion led to this study; to Mr. Gerald Winston, who aided in the statistical interpretation; and to Mrs. Mary Darby and Miss Beatrice McGlynn, who performed the abrasion tests.

Literature Cited

1. "A.S.T.M. Manual on Presentation of Data," Philadelphia, American Society for Testing Materials, April, 1943, p. 1.
2. Worthing, A. G., and Geffner, J., "Treatment of Experimental Data," New York, John Wiley and Sons, 1943, pp. 177 ff.
3. Davies, G. R., and Crowder, W. F., "Methods of Statistical Analysis in the Social Sciences," New York, John Wiley and Sons, 1933, p. 303.

Information for Contributors

to

Textile Research Journal

STYLE requirements for copy submitted to Textile Research Journal conform, in general, to the requirements of other scientific publications. This outline will clarify certain differences; it is also intended to assist new authors in their preparation of manuscripts. Observance of these requirements will be of material assistance to the editors.

Subject Matter

1. General. Papers for publication in the Journal should report the results of new research or of that which will add to existing information. Discussion of the work of others, on questions involved, should be confined to work which bears directly upon the author's point of departure, methods, arguments, or conclusions, and should be accompanied by literature citations. Authoritative and critical reviews are occasionally accepted, the decision to publish being based upon the value to research workers on the subject.

The Journal seeks to cover the broad field of scientific interest of the industry. To this end papers need not be confined to strictly textile subjects. They may be on the border line of pure science if they present information of value to researchers in textiles and their auxiliaries.

2. Experimental data should be included in sufficient detail to substantiate the conclusions which are drawn and also to enable others to reproduce the experiments. A concise treatment is important nevertheless. Frequently curves are sufficient without the inclusion of tabular data.

3. Letters to the Editor. Constructive comments and discussions of papers which have appeared in the Journal are solicited because such discussions stimulate interest in the subject and frequently are of material aid to progress in a given field.

4. Acceptance. Before papers are accepted for publication they are submitted to qualified persons for review, critical comment, and suggestions. Names of the reviewers are not disclosed except by their consent. The principal grounds for the rejection of papers are:

(*a*) That the subject matter is of too-limited reader interest.

(*b*) That the subject matter is not new.

(*c*) That the report indicates lack of thoroughness in the research upon which it is based.

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Manuscripts

1. Authors' Names and Business Connections. This information should be given exactly as the author desires to have it appear in the publication. The place where the work was done should be indicated. Subsequent change of address should be mentioned in a footnote.

2. Typing. Manuscript should be typed double-spaced on letter-size paper with generous margins. An original and a carbon copy should be submitted to the editor. Original photographs and drawings should accompany the original. Blue prints, photostats, or other reproductions may accompany the second copy.

3. Abstract. It is requested that a brief abstract (not over 150 words) accompany each paper.

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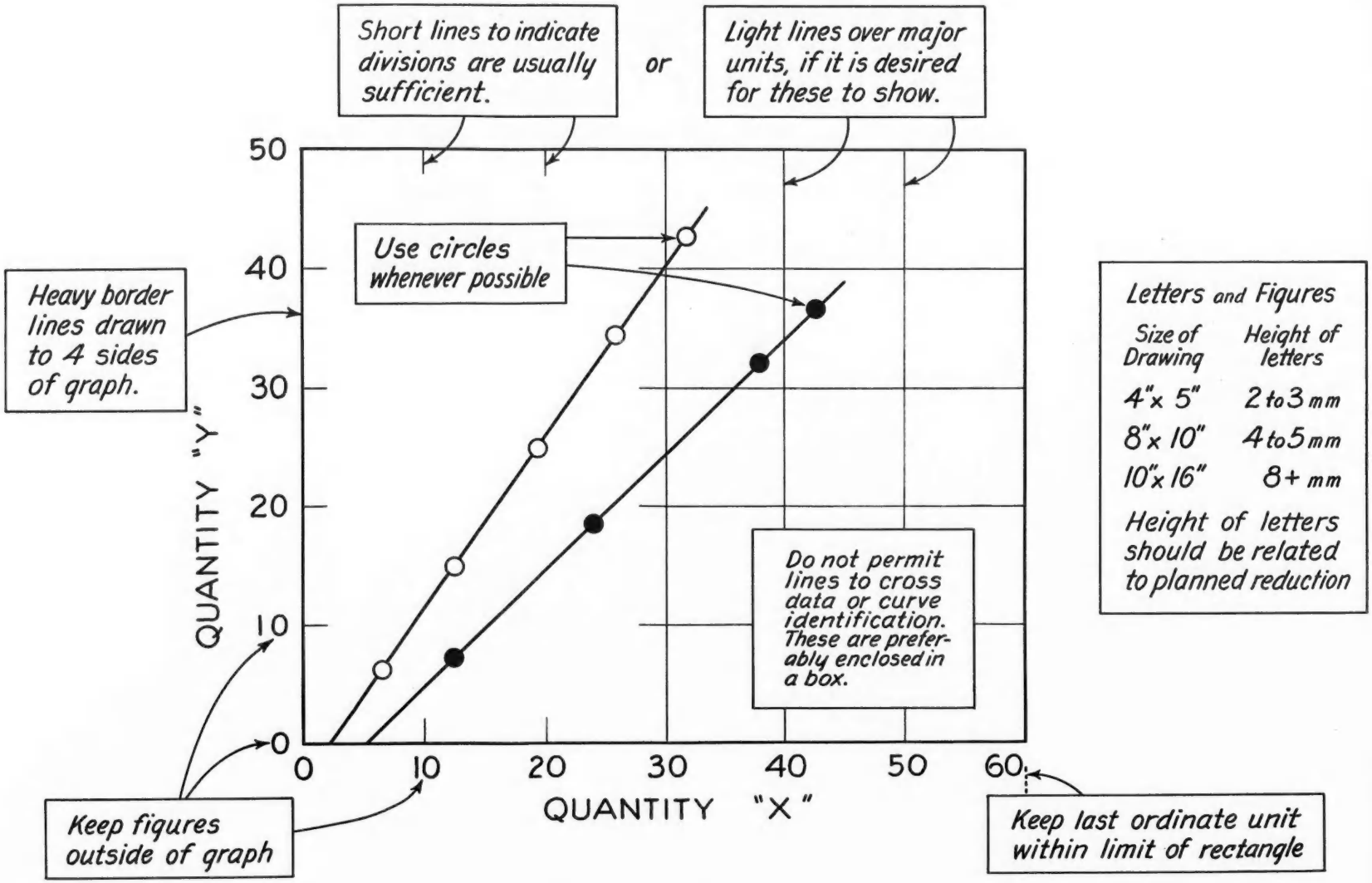
5. Equations. All equations should be verified by the author because alterations which require resetting of type are expensive. Exponents and subscripts should be legible and properly positioned. There should also be a clear distinction between small and capital letters, and there should be no


Short lines to indicate divisions are usually sufficient.
or
Light lines over major units, if it is desired for these to show.
Use circles whenever possible
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Do not permit lines to cross data or curve identification. These are preferably enclosed in a box.
Letters and Figures
Size of Drawing
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2 to 3 mm
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Height of letters should be related to planned reduction
QUANTITY "Y"
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0
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Keep figures outside of graph
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possibility of confusing the small letter "l" (el) with the number "1" (one), or the letter "O" with zero, etc.

Complicated structural formulas should be submitted in the form of India-ink drawings from which cuts can be made. Symbols and Greek letters should be carefully made.

6. Italics. Indiscriminate underlining of words in the text which the author desires to emphasize should be avoided. The JOURNAL rarely italicizes these but does italicize botanical names and the like. Also careless or non-uniform underlining of letters and symbols customarily set in italics should be avoided.

7. Drawings should be either on tracing cloth, coordinate paper ruled with blue lines (never orange or red), or should be the original drawings on draftsman's paper, in India ink. Clear photographic prints of black-and-white drawings are ordinarily acceptable. Rarely is it necessary to show the cross-section lines. Usually an indication of the main subdivisions, as shown on the accompanying diagram, is sufficient. When it is necessary to show the cross-section lines, green cross-section paper should be used, and the author should state his wish that they be reproduced.

The ultimate size of illustrations should be borne in mind when the drawings are planned. Most drawings can be reduced to the column width of the JOURNAL. Reference to the diagram should be made for the proper size of letters. Letters and figures made by stencils are preferable; they should never be typewritten. It is the style of the JOURNAL to place captions below each illustration; they should not appear on the drawings.

8. Photographs. Glossy prints of sharply focused photographs give the best results. Writing in ink on the back of a print or drawing may show on a cut. Since cracks and creases show in reproduction, pictures should be mailed flat, protected by adequate cardboard backing.

9. Tables. Every table should have a caption above it and be numbered in proper sequence with Roman numerals. Arrange columns to show pertinent data, with remarks (if any) concerning general conditions of experiment either as a footnote to the table or below the caption—e.g.:

TABLE I. RHEOLOGICAL PROPERTIES OF TEXTILE PRINTING PASTES

(Measurements on rotational viscometer: maximum rate of shear, 100 sec^{-1}; temperature, $30° \pm 0.1°C$)

For arrangement consult tables in current issues of the JOURNAL.

10. In *Literature Cited* data should be given in the following order: (1) authors' names followed by initials; (2) journal; (3) volume; (4) pages (inclusive); (5) month and year (in parentheses). If the number of references exceeds five, they should be listed in alphabetical order by name of author. If fewer than five, they may be arranged numerically, as reference to them appears in the text.*

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* An excellent article by A. J. Riker on the preparation of research manuscripts appeared in the November, 1946, issue of *Phytopathology*. This is a detailed discussion which includes a list of 28 references. The Publications Department has a limited supply of reprints of this article at 50 cents each.

Another very practical discussion by Florence E. Wall entitled "The Essentials of a Good Report," which was presented before the American Chemical Society in Chicago, September, 1946, was published in the *Journal of Chemical Education*, March, 1947.—*Editor*

ABSTRACTS

SERVICES AVAILABLE

Reprints of the original articles abstracted in TEXTILE RESEARCH JOURNAL *are often available from the publishers. Otherwise, it may be possible to secure copies of the periodicals. Orders should be sent direct to the publishers.*

Photostat negatives of articles abstracted will be furnished to members of the Institute at a cost of 25 cents per page for pages not exceeding 7 inches in width and 35 cents per page for larger sizes up to 11½ x 14 inches. Photostats of patents are 35 cents a page. Postage will be added.

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A list of periodicals, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given on page 37 of the January, 1947, issue.

ANALYSIS: TESTING: LABORATORY METHODS

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Saponification of Acetate

Some aspects of the saponification of acetate rayon. South Central Section, AATCC. *Am. Dyestuff Reptr.* **36**, P64–9 (Feb. 10, 1947).

Two types of saponification of cellulose acetate rayon (the surface or superficial type, and the diffuse type) may be obtained with all possible gradations between them. Cross sections have been prepared showing the sharp line of demarcation which can be obtained when the surface saponification is carried out. The effect of time, temperature, and concentration on the reaction rate when sodium hydroxide is used is also shown. A number of alkaline agents were tested at various pH values and temperatures. The type of saponification given by each is shown by dyeing the fibers and preparing cross sections. In general, it has been demonstrated that the saponification reaction can be controlled to give the desired results with a minimum of cost and effort providing proper care is exercised in selecting the saponifying agent and the conditions for the reaction.

Text. Research J. May 1947 Authors

Absorption of Liquor by Cellulose

Absorption of mercerizing liquor by cellulose in sheet form. Olof Samuelson. *Svensk Papperstidn.* **50**, 21–5 (Jan. 31, 1947).

The rate of absorption of caustic soda solution by sheets of rayon pulp has been studied in the laboratory. The time (t) for different heights of absorption (h) was determined at constant liquid level. The curves represented by $h = f(t)$ vary considerably for different pulps and in some cases even cross each other. A discussion of the significance of the rate of absorption with reference to the industrial utilization of the cellulose leads to the conclusion that the conventional method of determining the time of absorption for a rise of 50 mm., for instance, has comparatively little value. A much lower figure should be chosen—say, 10 or 15 mm. Study has been made of the effect of temperature and concentration of the steeping liquor as well as of the content of hemicellulose in the liquor. In the last section of the paper the author reports investigations concerning variations in the composition of the alkali cellulose in different parts of the sheet. These experiments were carried out in the laboratory as well as in mill operation. It is shown that such variations can depend upon overdrying of the pulp.

Text. Research J. May 1947

Color Specifications

Interrelation of color specifications. Dorothy Nickerson. Mimeographed Bulletin, U. S. Dept. of Agriculture, Production and Marketing Administration, Cotton Branch. 45 pages.

The interrelation of color specifications for standard reference material is discussed in 3 sections. Section 1 relates to various ways of graphing or transforming I.C.I. data: (x, y)-diagram; Judd U.C.S. diagram (1932–5); MacAdam U.C.S. diagram (1937); Breckenridge and Schaub rectilinear U.C.S. (1939); Scofield (1940); Adams (1940); Moon and Spencer (1943); Saunderson and Milner (1944). Section 2 lists several sets of standard reference material by which conversions may be made, or for which data in terms of specifications recommended in ASA-Z44 standard are available in whole or in part: dominant wave length—

purity; Munsell; ISCC-NBS; Ostwald; TCCA standards; Maerz and Paul *Color Dictionary*; Ridgway; Lovibond glasses; Wratten filters. Section 3 reviews the problem of color-tolerance specification as it relates to interrelation of color specifications, and includes a summary table of formulas developed for tolerance work. (This paper was given as part of a Symposium on Color at a joint meeting of the Inter-Society Color Council and the Technical Association of the Pulp and Paper Industry, held in New York, Feb. 25, 1947.)

Text. Research J. May 1947

Deterioration of Flameproofed Fabrics

The permanence of flameproofed fabrics. S. A. Rulon, M. J. Sostmann, and L. I. Phillips. *Am. Dyestuff Reptr.* **35**, 489–92, 501–2 (Oct. 21, 1946).

Tests on untreated fabrics and fabrics flameproofed with antimony oxide and chlorinated paraffin indicated that: (1) strength loss on heating, up to about 115°C, is due principally to breakdown of the cellulose itself; (2) similarly, in artificial weathering, photochemical effect on the cellulose is of greater importance than attack on the cellulose by decomposition products of the finishing compound; (3) the combined degrading effect of water and unfiltered arc irradiation is far greater than that of the irradiation alone; and (4) industrial E carbons, having a high intensity in the infrared region of the spectrum, were effective in producing degradation in the treated fabrics, especially when a pigment having high infrared absorption, e.g., lampblack, was used. In accounting for the difference in extent of degradation in the top as compared to the lower rack in the weathering unit, a figure showing the intensity distribution in a vertical plane around the Sunshine carbon arc is reproduced; the lower rack receives a smaller proportion of the ultraviolet from 2,700–3,200 Å, and a larger proportion of the radiation from 3,000-8,000 Å. K. S. Campbell

Text. Research J. May 1947

Toxicity of Germicides

Toxicity of germicides. L. C. Barail. *Am. Dyestuff Reptr.* **35**, 521–4 (Nov. 4, 1946).

Informative tests, which are made before performance tests in the testing of germicides for application to fabrics, include an animal injection test for toxicity, a minimum lethal dose test, an intradermal injection test for skin irritants (on rabbits), and patch tests for primary skin irritants and for cutaneous sensitization, on humans. A tolerance of 1 to 2% in the latter 2 tests is recommended, as a certain irritation of allergic nature is to be expected. In tests on 250 compounds, only 15 were found suitable for use on fabrics and therefore submitted to toxicity tests. Out of these 15 only 7 were nontoxic. (The criterion here was that if 1 cc. of the normal concentration of germicide injected into a mouse caused death within 3 days, it would endanger the life of infants who might suck their garments.) For thirteen the minimum lethal dose was high enough to make them safe under normal conditions of use. Only 6 did not cause any irritation on animals; 3 of these were insoluble in water and therefore inconvenient to apply to fabrics, and of the remaining 3 one was an irritant to human skin and one a skin sensitizer. One which was nonirritating and nonsensitizing was a long-chain phenyl-mercury derivative, lactoxyphenyl-mercuric ammonium lactate. K. S. Campbell

Text. Research J. May 1947

Moisture Determination

A new electronic hygrometer. Anon. *Dyer* **97**, 178–80 (Feb. 14, 1947).

A description is given of an electronic hygrometer which is designed for continuous determination of the moisture content of cloth. The instrument is said to be capable of detecting variations of a fraction of 1.0% in moisture content down to zero moisture. J. A. Woodruff

Text. Research J. May 1947

Sizing

Permanent sizing approved by test in Quartermaster Project at I.T.T. Anon. *Textile World* **97**, 122–3 (Feb. 1947).

A report on test of various permanent warp-sizing materials to replace starch made by the Institute of Textile Technology for the Office of The Quartermaster General. Of 44 materials tested, 4 were found to have satisfactory mildew-resistant qualities: Dow's Carboxymethocel A (CMC-A); Monsanto's Stymer; Rohm & Haas' RHoplex MR; and American Cyanamid's Aerotex 625. Test procedures are given; also a table showing breaking strength and elongation of yarns which have been sized with these 4 materials as well as with starch, as compared with those of unsized yarns.

H. J. Burnham

Text. Research J. May 1947

Moscrop Tester

A modification of the Moscrop yarn tester. J. G. Martindale and B. Hogley. *J. Text. Inst.* **37**, T281–4 (Dec. 1946).

The tester was geared down to a traverse speed of 1 ft./min. This led to a disadvantage because both jaws were open with the carriage "in," and control of the yarn was lost for a sufficient length of time to permit untwisting. This was rectified by a simple addition to the driving mechanism which caused the carriage speed to be increased to 14 ft./min. during the return motion. With these attachments, the testing speed is 480 tests in 90 min. A. R. Martin

Text. Research J. May 1947

Compressibility of Wool

Note on the compressibility of wool. C. M. van Wyk. *J. Text. Inst.* **37**, T285–92 (Dec. 1946).

A relationship between pressure and volume for a mass of wool fibers is derived on the assumption that the compression of the mass consists solely of the bending of the fibers. It is suggested that com-

pressibility measurements constitute a convenient method for comparing the flexural elastic properties of the fibers composing different wool samples. Authors

Text. Research J. May 1947

Friction of Wool

Some measurements of the friction of wool and mohair. A. J. P. Martin and R. Mittlemann. *J. Text. Inst.* **37**, T269–80 (Dec. 1946).

An apparatus constructed for measuring the friction of fibrous material and its use are described. It was found desirable to work with single fibers. Measurements on wool and mohair under a variety of conditions are given, for both directions of slip. The coefficient of friction of wool and mohair decreases with increasing load. A logarithmic relationship has been found to hold approximately for the μ_{T-R} of wool against wool. For wool and mohair, the coefficient of friction is positively correlated with the diameter of the fibers.

Authors

Text. Research J. May 1947

Vegetable Matter in Wool

The sodium-hydroxide method for the determination of vegetable matter in wool. G. C. Le Compte and M. R. Coe, Jr. *Am. Dyestuff Reptr.* **35**, 509–10 (Nov. 4, 1946).

A method for determining vegetable matter in wool consists of adding 400 ml. of boiling 10% NaOH to a 20-g. sample of wool, allowing the sample to stand for 2 or 3 min., then diluting it with approximately 500 ml. cold water, filtering off the vegetable matter, washing first with water, then with 5% NaOCl, again with water, then drying and weighing it. The method is quick and fairly accurate, and, together with the ordinary extractives and ash tests, approximates rather well the quantity of hard taggy matter found in some samples of scoured wool. Authors

Text. Research J. May 1947

CHEMICAL AND PHYSICAL RESEARCH

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Oxidation of Cellulose

The oxidation of cotton cellulose by solutions of potassium permanganate in the presence of a reduced vat dye. D. N. Mehta and H. A. Turner. *J. Soc. Dyers and Colourists* **63**, 15–20 (Jan. 1947).

The results obtained during the accelerated oxidation of cotton cellulose by solutions of sodium hypochlorite in the presence of leuco-Cibanone Orange R lead to a hypothesis that vigorous and characteristic acceleration will not take place in a solution of oxidizing agent the corrected potential of which is below +1,484 mv. For the purpose of testing this hypothesis the effect of dilute solutions of potassium permanganate on dyeings of leuco-Cibanone Orange R on cotton has been investigated at different pH values. The corrected potential of these solutions did not attain the critical value +1,484 mv. when measured at 20°C, and the accelerating action of the leuco-dye on cellulose oxidation by these solutions was very small. An increase in the corrected potential was not obtained by an increase in working temperature. Results obtained, although not inconsistent with the hypothesis stated above, do not furnish positive confirmation of it.

Text. Research J. May 1947 Authors

Recrystallized Cellulose

On the recrystallization of amorphous cellulose. P. H. Hermans and A. Weidinger. *J. Am. Chem. Soc.* **68**, 2547–52 (Dec. 1946).

Amorphous cellulose may be prepared by milling very dry ramie, rayon, or wood pulp in a vibrating ball mill from 1 to 4 hrs. The product shows no fibrillation and is soluble in 1*N* NaOH. Its x-ray photograph shows a broad band whose maximum intensity lies at the site of the A_3 interference of cellulose *II*. If a sample of the amorphous cellulose is placed in a thin-walled capillary, wetted with water, sealed, then heated to 100°C, reopened and allowed to dry, the sample will give the x-ray photograph of crystalline cellulose *II* (hydrate). Computation from the intensities of the cellulose *II* in the x-ray photograph and data on the comparative heats of wetting of the material before and after recrystallization indicate that the recrystallized material contains 60–70% amorphous cellulose.

A. R. Macormac

Text. Research J. May 1947

Cellulose Transformations

On the transformation of cellulose II into cellulose IV. P. H. Hermans and A. Weidinger. *J. Colloid Sci.* **1**, 495–504 (Dec. 1946).

The transformation of mercerized ramie fibers into cellulose IV (high-temperature cellulose) by heating the alkali cellulose in glycerin is accelerated by the alkali only because its swelling effect causes better penetration of the glycerol into the cellulose. X-ray data on cellulose IV are given. E. D. Klug

Text. Research J. May 1947

Structure of Cellulose Powder

Electron microscope structure of cellulose powder from wood pulp ground in a very dry condition. P. H. Hermans. *J. Am. Chem. Soc.* **68**, 2730–2 (Dec. 1946).

Wood-pulp fibers were ground for 5 hrs. in a vibrating ball mill and then dispersed in water by ultrasonic radiation (200,000 cycles). The particles ranged from 1 to 10μ. This material contained less than 3% ash. These powders were permitted to recrystallize from water. Electron micrographs (6,400 to 20,000×) of the original and recrystallized material did not show any fibrillar structure nor was there any evidence of the elementary fibrils of Hess, but the electron micrographs did show rather compact ag-

gregates whose actual structure was difficult to recognize.

A. R. Macormac

Text. Research J. May 1947

Detection of Oxycellulose

Aromatic mono- and diamines as specific reagents for the detection of oxycellulose. Matiya Kraïchinovich and Mladen Kraïchinovich. *J. Applied Chem.* (*U.S.S.R.*) **19**, 420–3 (1946) (through *Chem. Abstr.* **41**, 1434*f* (Mar. 10, 1947)).

It was shown that organic amines can be bound to the CO_2H groups of oxycellulose. Diamines (benzidine, *p*-$(NH_2)_2C_6H_4$, *m*-toluenediamine, chloro-*m*-phenylenediamine) in alcohol solution were boiled with the cellulose samples, diazotized *in situ*, and coupled with various naphthols, naphthylamines, and resorcinol, to yield color tests ranging from pale yellow to red-violet. Similar colors are produced when the cellulose is first treated with amine and treated with various diazonium solutions.

Text. Research J. May 1947

Determination of Oxycellulose

Determination of carbonyl number as a new chemical constant of oxycellulose. Matiya Kraïchinovich and Mladen Kraïchinovich. *J. Applied Chem.* (*U.S.S.R.*) **19**, 424–7 (1946) (through *Chem. Abstr.* **41**, 1434*g* (Mar. 10, 1947)).

An analytical method is presented by which separate estimation of oxycellulose is made as distinct from hydrocellulose; it is based on the hydroxylamine reaction with the CO groups of oxycellulose. The "carbonyl number" thus obtained can be used, together with the Cu number, as a measure of cellulose degradation. One g. of sample is wetted with 20 cc. of 0.1*N* $NH_3(OH)Cl$ and 10 cc. of 0.1*N* NaOH and allowed to stand for 20 min. at room temperature, after which the mixture is titrated with 0.1*N* HCl with methyl orange indicator to determine the amount of hydroxylamine in the residue. The carbonyl number is calculated as $2.8\ (a-b)/c$, where a is cc. of 0.1*N* HCl in blank titration, b is cc. of 0.1*N* HCl used in the unknown titration, and c is the weight of cellulose in g.

Text. Research J. May 1947

Chlorination

Chlorination by aqueous sodium hypochlorite. C. Y. Hopkins and Mary J. Chisholm. *Can. J. Research* **24B**, 208–10 (1946) (through *Chem. Abstr.* **41**, 734*c* (Feb. 10, 1947)).

Nuclear chlorination of cyclic compounds is more widespread than had been supposed previously. The compound is dissolved or suspended in dilute alkali, mixed with NaOCl solution, and left for 1 hr. at ordinary temperature with occasional stirring. Suspended Ca hypochlorite gives lower yields. The following derivatives were prepared (% yield in parentheses): 3-chloroanisic acid (80), 5-chlorovanillic acid (85), 3,5-dichloro-4-hydroxybenzoic acid (50), 6-chloro-3-methoxybenzoic acid (60), *5-chloro-2-hydroxy-4-acetoxybenzoic acid*, m. 176–7° (uncor.) (40), 6-chloropiperonylacetic acid (80), 3-chloro-4-methoxyphenylacetic acid (50), 5-chlorovanillin (90), 6-chloropiperonal (poor), 5-chloro-2, 4-dihydroxy benzaldehyde (80), 2,4-dichlorophenoxyacetic acid (75–80), 4-chloro-2-methylphenoxyacetic acid (85).

Text. Research J. May 1947

Deformation

Deformation in relation to time, pressure, and temperature. P. G. Nutting. *J. Franklin Inst.* **242**, 449–58 (Dec. 1946).

By separating deformation into the 3 stages (instantaneous yield, a slower yield which is a period of readjustment, and, finally, a steady state) which always characterize it, and by analyzing separately the dependence of deformation on time, temperature, and pressure, equations are derived which are in good agreement with data of many types. Thermodynamic relations involving changes in energy during deformation are also derived. The various equations are applied to data on yield and temperature changes for steel tape. W. E. Davis

Text. Research J. May 1947

Lignin

The molecular weight of lignin. Nils Gralen. *J. Colloid Sci.* **1**, 453–63 (Oct. 1946).

Lignin obtained by heating wood with thioglycollic acid is heterogeneous and has an average molecular weight of about 7,000 as determined from sedimentation and diffusion data. Since analysis indicated that the lignin had the formula $C_6H_9O_3$—OCH_3, the average degree of polymerization (D.P.) was 36. Treatment of thioglycollic acid lignin with more thioglycollic acid gave more homogeneous lignins of about half the D.P. The author believes that the unit of molccular weight 3,500–4,000 is probably more stable. The axial ratio of the molecule was found to be proportional to its molecular weight. For a lignin of molecular weight 7,000, the ratio is 5. Thioglycollic acid lignins prepared from different plants have about the same molecular weight. Alkali lignins and hypobromite lignin have about the same molecular weight (7,000) as thioglycollic acid lignin. Acetic acid lignin has a higher molecular weight as have thioglycollic acid lignins which have been pretreated with acid or base. It is suggested that the higher molecular weight is due to condensation.

Text. Research J. May 1947 E. D. Klug

Theory of Substitution in Naphthalenes

A theoretical discussion of further substitution in disubstituted naphthalenes. Part II—Heteronuclear mononitronaphthylamines. H. H. Hodgson and D. E. Hathway. *J. Soc. Dyers and Colourists* **62**, 241–2 (Aug. 1946).

Substitution by diazo-coupling, halogenation, nitration, and mercura-

tion of 5-, 6-, and 8-nitro-1- and -2-naphthylamines is discussed from the standpoint of the Vesely and Jakes quinonoid hypothesis of substitution modified by modern electronic and resonance theory. All the apparent anomalies are adequately explained by the modified treatment. Authors

Text. Research J. May 1947

Deformation of Polymers

Dependence of the average transversal on the longitudinal dimensions of statistical coils formed by chain molecules. Werner Kuhn. *J. Polymer Sci.* **1**, 380–8 (Oct. 1946).

The transversal dimension of the statistical coil formed by a long-chain molecule in solution or in bulk rubber is independent of the longitudinal dimension, h_1, of the coil as long as h_1 is small compared with the length of the stretched molecule. On the other hand, the lateral dimensions of a coil whose longitudinal vector, h_1, is oriented in the direction of stretch undergoes a forced contraction during the deformation of bulk rubber. The forced contraction of the transversal dimension is therefore supplying an extra contribution to the entropy change the amount of which has been estimated in earlier work. This change influences the numeral factor occurring in the formula connecting the strain-stretch relationship for rubber with the molecular weight of the net fragments. Author

Text. Research J. May 1947

Viscosity of Polymer Solutions

The intrinsic viscosity of polymer solutions. P. Debye. *J. Chem. Phys.* **14**, 636–9 (Oct. 1946).

A mathematical picture is given for the motion of a polymer molecule and its parts, showing why Staudinger's rule relating intrinsic viscosity of polymer solutions and the molecular weight of the polymer holds. R. W. Eyler

Text. Research J. May 1947

Polymerization Mechanism

The photochemical polymerization of methyl vinyl ketone vapor. T. T. Jones and H. W. Melville. *Proc. Roy. Soc.* **A187**, 19–36 (Oct. 8, 1946).

The photo-polymerization of methyl vinyl ketone vapor at pressures up to 60 mm. and temperatures between 0° and 60°C has been investigated with the following results: the kinetic order of the reaction increases with pressure and decreases with increasing temperature; the highest order recorded is 7.4; the temperature coefficient of the reaction is negative, but the rate does not obey the usual exponential relationship; the polymer produced is insoluble. These observations all point to the intervention of a branched chain mechanism, and on this basis the abnormal kinetics may be quantitatively explained. The polymerization occurs by the splitting of the methyl vinyl ketone molecule into free radicals, probably $CH_3 + CH_2{=}CH \cdot CO$. The former radical initiates a free radical polymerization which is terminated by a mutual interaction of two radicals in the vapor phase. The $CH_2{=}CH \cdot CO$ radical appears to break up with the formation of carbon monoxide, the $CH_2{=}CH$ radicals combining to yield butadiene, which plays no further part in the reaction. A micro-vapor pressure analyzer is described which will deal with a few cubic mm. of vapor at normal temperature and pressure. Authors

Text. Research J. May 1947

The free radical polymerization of the vapors of certain vinyl derivatives. T. T. Jones and H. W. Melville. *Proc. Roy. Soc.* **A187**, 37–53 (Oct. 8, 1946).

The free radical polymerization of the vapors of vinyl chloride, acrylic nitrile, styrene, butadiene, and methyl isopropenyl ketone has been investigated using methyl radicals from photo-decomposing acetone. Although all the reactions do not exhibit ideal behavior, it is possible by the application of kinetic methods to measure the ratio of the propagation to the termination coefficients for these polymerization reactions. In this way relative values of the quantitative tendency for a molecule to polymerize by a free radical mechanism can be computed. Authors

Text. Research J. May 1947

Polyvinyl Alcohol

Nature of the carbonyl groups in polyvinyl alcohol. John T. Clark and Elkan R. Blout. *J. Polymer Sci.* **1**, 419–28 (Oct. 1946).

The rapid drop in the viscosity of polyvinyl alcohol solution to a constant value in the presence of $0.02N$ HCl may be due to the presence of a cross-link between polymer chains which is destroyed by the acid. The presence of 0.4 mole % of carbonyl in the polymer was shown both spectrophotometrically and by the use of the *p*-nitrophenylhydrazine derivative. The carbonyl content appears to be independent of the chain length and to be ketonic. It is likely that cross-links in the polymer are due to ketals formed between chains.

Text. Research J. May 1947 E. D. Klug

Derivatives of Polyvinyl Alcohol

Water-soluble alkoxyacetals of polyvinyl alcohol. K. F. Beal and C. J. B. Thor. *J. Polymer Sci.* **1**, 540–8 (Dec. 1946).

Acetals of polyvinyl alcohol (PVA) and the following aldehydes were prepared: methoxyacetaldehyde, ethoxyacetaldehyde, 2-methoxyethoxyacetaldehyde, 2-ethoxyethoxyacetaldehyde, 2-*n*-propoxyethoxyacetaldehyde, tetrahydrofurfuryloxyacetaldehyde and 3-methoxypropionaldehyde, or with mixtures of these aldehydes. The resulting thermoplastic resins are soluble in organic solvents if the extent of reaction is above 80% of the PVA. Where the extent of reaction is appreciably less than 80%, products are also soluble in cold water and become water-insoluble at temperatures which vary with the aldehyde used and decrease with the extent of re-

action. Films of some of these products compare favorably with polyvinyl butyral in strength.

Text. Research J. May 1947 E. D. Klug

Solvation of Particles

Dependence of solvation on particle shape. B. Jirgensons. *J. Polymer Sci.* **1**, 475–83 (Dec. 1946).

Solvation is dependent on the surface area of particles and on the chemical relationship between solvent and solute. True chemical solvation is very small and there is no reason to believe that there is a thick, tightly held solvent layer. No good relationship between solvation and viscosity exists. The stability of lyophilic colloids, especially proteins, depends very little on solvation; it is more dependent on the groups which are formed on the surface of particles. Methods of measuring solvation are discussed. No new data. E. D. Klug

Text. Research J. May 1947

Starch

Chlorine-treated starch. Robert Haller. *Helv. Chem. Acta* **29**, 1688–9 (Dec. 2, 1946) (in German) cf. *B.I.P.C.* **17**, 95 (through *Bull. Inst. Paper Chem.* **17**, 263 (Jan. 1947)).

No morphological changes and only slight changes in paste formation were noted in potato starch which had been treated twice with cold saturated chlorine water. Fehling solution was not reduced, but Ciba yellow GN was reduced in alkaline medium. Potassium iodide gave a blue-violet color. The washed starch, when treated with silver nitrate and irradiated, became dark gray, indicating adsorbed chlorine. Treatment of starch at 40–50°C with chlorine water gave a product completely lacking paste-forming properties, but which was otherwise similar to starch treated in the cold. In general, the halogens (fluorine has not been investigated) are first adsorbed by the starch, whereupon mild degradation occurs, but not to the point of the so-called "soluble starches."

Text. Research J. May 1947

Stress Distribution in Solids

The distribution of stress in the neighborhood of a crack in an elastic solid. I. N. Sneddon. *Proc. Roy. Soc.* **A187**, 229–60 (Oct. 22, 1946).

The distribution of stress produced in the interior of an elastic solid by the opening of an internal crack under the action of pressure applied to its surface is considered. The analysis is given for "Griffith" cracks and for circular cracks, it being assumed in the latter case that the applied pressure varies over the surface of the crack. For both types of crack the case in which the pressure is constant over the entire crack surface is considered in some detail. The stress components are tabulated and the distribution of stress is shown graphically. The effect of a crack (of either type) on the stress produced in an elastic body by a uniform tensile stress is considered and the conditions for rupture are deduced.

Text. Research J. May 1947 Author

Sugar-Aldehyde Reaction

Reaction between sucrose and aldehydes. Ippolito Sorgato. *Ann. chim. applicata* **33**, 113–17 (1943) (through *Chem. Abstr.* **41**, 717*g* (Feb. 10, 1947)).

By condensation of sucrose with HCHO at 80–120°, there is formed a solid polymerized compound which is transparent, slightly yellow, and insoluble in H_2O and organic solvents. In the intermediate reactions, hemiacetals are formed; these are transformed into acetals with 2 methylene bridges between 2 molecules of hexose.

Text. Research J. May 1947

Terephthalic Acid

Terephthalic acid. C. F. Koelsch. *Org. Syntheses* **26**, 95–7 (1946) (through *Chem. Abstr.* **41**, 738*i* (Feb. 10, 1947)).

Details are given of the oxidation of *p*-MeC_6H_4Ac with HNO_3 and then with $KMnO_4$, the yield of *p*-$C_6H_4(CO_2H)_2$ being 84–8%.

Text. Research J. May 1947

Elastic Properties of Yarns

Elastic properties of textile yarns, represented with the aid of a mechanical model. H. A. Vreedenberg. *J. Polymer Sci.* **1**, 329–39 (Oct. 1946).

Some measurements of the elastic behavior of cellulose acetate, viscose rayon, and natural silk are presented. The results are described by means of a mechanical model having 7 independent fundamental constants. It is necessary to introduce the possibility of a blockade of part of the model. The values for the constants associated with the blockade can be determined experimentally; they are found to be independent of the moisture content. This blockade may represent the formation of new points of attachment between molecular chains during the increase in extension. E. D. Klug

Text. Research J. May 1947

Chroming of Wool

The dyeing of wool with chrome dyes. Part 2. The chroming of wool. P. W. Carlene, F. M. Rowe, and J. B. Speakman. *J. Soc. Dyers and Colourists* **62**, 329–38 (Nov. 1946).

An interesting feature of the titration curve of wool with chromic acid is that it lies far to the right of the hydrochloric acid curve on the pH scale, indicating that the anions of chromic acid have a marked affinity for wool. In boiling potassium dichromate solutions $HCrO_4^-$ ions are rapidly absorbed, a corresponding number of hydrogen ions combine with the acid side-chains of the wool, the pH of the solution rises, and disulfide bond attack is likely to be severe. Presence of ammonium sulfate minimizes the rise in pH. As expected, it was found that this agent reduced disulfide-bond hydrolysis, as evidenced by a decreased rate of reduction of hexavalent to trivalent chromium; possibility of direct attack on disulfide bonds could not, however, be excluded. In a study of the elastic properties of chromed fibers before and after removal of the chromium, no evidence was obtained that chro-

mium-containing cross-linkages are formed between the peptide chains of wool during mordanting. The tentative conclusion was reached that chromium compounds are mechanically deposited rather than chemically combined in the wool fiber. K. S. Campbell

Text. Research J. May 1947

Hand of Wool

The handle of wool. E. H. Mercer and K. Rachel Makinson. *J. Council Sci. Ind. Research* **19**, 200–1 (1946) (through *Chem. Abstr.* **41**, 290*i* (Jan. 10, 1947)).

One of the factors influencing the handle of wool is the coefficient of friction (μ) of the component fibers. Impaired handle following treatment with alcoholic KOH solution is correlated with an increase in μ. Softening agents improve the handle by reducing μ.

Text. Research J. May 1947

BLEACHING: DYEING: FINISHING

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Bleaching of Jute

Permanent bleaching of ligno-cellulosic materials. P. L. D. Peill. *Nature* **158**, 554 (Oct. 1946).

Photographs of jute, both untreated and acetylated, are given, showing that discoloration due to light has been reduced to an imperceptible amount by the acetylation treatment. A tentative explanation is given to the effect that the discoloration is caused by polymerization under the influence of light of the phenolic groups of the lignin to colored quinones; reaction between the phenolic groups and alkylating or acetylating reagents prevents the discoloration.

Text. Research J. May 1947 R. W. Eyler

Detergents

A study in detergency. Northern New England Section, AATCC. *Am. Dyestuff Reptr.* **36**, P91–6 (Feb. 24, 1947).

A study of detergents for wool has been made with the use of the Detergency Comparator developed by the Wool Detergency Subcommittee of the AATCC. The Comparator is a miniature wool dolly washer made to scale from the usual mill washer. An anionic, a cationic, and a non-ionic-type synthetic detergent were selected for comparison with a red oil soap of 95% soap content and 10-degree titer. Comparisons were made without assistants, and with sodium sulfate, sodium carbonate, sodium bicarbonate, and sulfuric acid. The stock was oiled with a 50–50 mineral oil-grease oil blend to which lampblack was added to permit evaluation of scouring efficiency by reflectance measurements with the G. E. Recording Spectrophotometer; also the amount of oil remaining after scouring was determined by Soxhlet extraction. Synthetic detergents showed excellent results in removal of this type of oil but were not equal to soap for lampblack removal; sodium carbonate was the best of the assistants tested. K. S. Campbell

Text. Research J. May 1947

Theory of Color in Dyestuffs

Color and structure of indigo, indanthrene, and similar compounds discussed from the standpoint of modern resonance theory. H. H. Hodgson. *J. Soc. Dyers & Colourists* **62**, 176–8 (June 1946).

A discussion is given on the structural grouping responsible for the color of indigo, indanthrene, phenylene blue, and other dyes as suggested by the application of resonance theory. 19 references.

J. A. Woodruff

Text. Research J. May 1947

Color Lakes

A review of the literature on color lakes. W. B. Blumenthal. *Am. Dyestuff Reptr.* **35**, 529–38, 543–5 (Nov. 18, 1946).

The literature on lakes is comprehensively reviewed, summarizing all the theoretical and practical aspects of pigment-lake technology. The term "lakes" is taken in the broad sense and hence, since the fields of lake-making and dyeing overlap, the latter subject is also included to a considerable extent. 555 references and a lengthy subject-and-name index are appended.

K. S. Campbell

Text. Research J. May 1947

Coprantine Dyestuffs

Practical experience with Coprantine dyestuffs. F. Herndl. *Ciba Rev.* **51**, 1857 (1946) (through *Chem. Abstr.* **41**, 1105*i* (Feb. 20, 1947)).

For dyeing with Coprantines the dye must be completely dissolved at the start of the operation. The use of soda ash and Albatex PO together in the dye bath is recommended. The dye bath should be exhausted thoroughly before the addition of the Coprantine salt. The amount of glauber's salt in any particular dyeing operation depends on the length of the dye bath and the depth of the shade to be dyed. For pale shades, especially when the short dye bath has been used, the glauber's salt may be omitted completely; dark shades, such as navy blues and dark browns, require an addition of 40–60% of glauber's salt. Directions are given for shading to pattern. Good reserves on fabrics containing acetate rayon are obtained only if the fiber is lustrous, and then only when Albatex PO is present in the dye bath. Saponification, even a partial saponification, of the acetate rayon leads to unsatisfactory reserves, and addition of soda to the dye bath should be avoided. Suitable products for use in producing acetate reserve styles are Coprantine Yellow GRLL, Violet Brown BL, Grey GGL, Orange 2BRL, Bordeaux BGL, Blue GLL, RLL, and Green G.

Text. Research J. May 1947

Dye-Chromium-Fiber Complex

The nature of the dye-chromium-fiber complex in the case of wool dyed with certain chrome mordant dyes. E. Race, F. M. Rowe, and J. B. Speakman. *J. Soc.*

Dyers and Colourists **62**, 372–83 (Dec. 1946).

The constitutions of the dye-chromium complexes formed when 3 mordant dyes were applied to wool by the 3 common chroming methods were established. The technique employed was: (1) the wool was dyed with a small amount of dye to complete exhaustion; (2) the amount of excess chromium in the wool was determined by extraction with oxalic acid; and (3) the residual amount of chromium was related to the known amount of dye on the fiber. In the case of the 3 dyes examined it was found that a single pure complex in which 2 molecules of dye are associated with 1 atom of chromium was produced. The only possible combinations of complex and fiber appear to be salt linkages between chromium and sulfonic groups in the complex and amino-groups in the fiber; thus the complex formed in the fiber is in effect an acid dye of high molecular weight. The increased fastness can be attributed to the increase in molecular size and consequent increase in van der Waals forces binding it to the fiber, and to the large size of the lake molecules, formed *in situ*.

K. S. Campbell

Text. Research J. May 1947

Acetate Dyeing

Celanese-Fortisan dyeing and printing. A Mellor. *J. Soc. Dyers and Colourists* **62**, 168–73 (June 1946); *Am. Dyestuff Reptr.* **35**, 348–52 (July 15, 1946).

The dyeing of cellulose acetate fabrics is reviewed under the headings of "plain and crepe fabrics," "dyeing for discharge printing," and under "discharge printing" alone. Some comment is made on finishing procedures as well as on gas-fume fading. A method of applying acid dyes to cellulose acetate fabrics by padding with alcohol, water, and sodium thiocyanate is given. General information is offered on the dyeing and printing of Fortisan.

J. A. Woodruff

Text. Research J. May 1947

Cotton Piece Goods Dyeing

Cotton piece goods dyeing in the past 25 years. R. E. Rupp. *Am. Dyestuff Reptr.* **35**, 650–2 (Dec. 2, 1946).

A review.

Text. Research J. May 1947

Garment Dyeing

Some suggestions on garment dyeing. W. J. Macnab. *Fibres, Fabrics & Cordage* **13**, 420, 422–4, 426 (1946) (through *Chem. Abstr.* **41**, 1844*f* (Mar. 20, 1947)).

Problems of dyeing mixed fabrics and finished garments are discussed and numerous suitable dyes are listed.

Text. Research J. May 1947

Dyeing Properties of Synthetics

Preparation, dyeing, and finishing of the new fibers. C. C. Wilcock. *J. Soc. Dyers and Colourists* **63**, 41–5 (Feb. 1947).

A summary of the more recent trends in the methods used for the preparation, dyeing, and finishing of rayons. General information is given on the dyeing properties of rayons, including nylon, Vinyon, casein fiber, Rayolanda, and the alginates. Author

Text. Research J. May 1947

Dyeing Properties of Viscose

Dyeing properties of some viscose rayons. J. Boulton and J. Wardle. *J. Soc. Dyers and Colourists* **63**, 8–14 (Jan. 1947).

Principal dyeing differences among different rayons are: (1) increased dyeing rate due to increased surface with decrease in filament denier; (2) decreased dyeing rate due to smaller average pore size in the case of highly oriented, high-tenacity yarns; (3) differences in dyeing rate due to variations in conditions of spinning, especially composition and temperature of the spinning bath and method of drying the yarn, resulting in different ratios of tough "skin," resistance to dye diffusion, to the amorphous, more readily penetrated "core"; (4) optical behavior which requires that for optical similarity the amounts of dye used must be in inverse relation to the cross-sectional areas of the filaments. Durafil (very highly oriented, with a filament denier of 0.3) sometimes may not be dyed to match normal viscose because it is so strongly dichroic that almost all the light vibrating in one polarized plane is transmitted and is not absorbed by the dye at all; vat and developed dyes, however, overcome this effect because they exhaust well and are not dichroic, and therefore do not lose apparent depth on Durafil. Diffusion rates and half-dyeing times plotted against filament denier for 3 common direct dyes on 19 viscose rayons with filament deniers ranging from 0.3 to 9.3 showed that in general the yarns behave consistently regardless of the dye used. Furthermore, the swelling action of the hot dye bath was shown to be essentially the same for all viscose rayons. Differences in viscose dyeing behavior do show up in the dyehouse, but not to a larger extent than those for other fibers and probably give less trouble than neppy cotton, lousy silk, or tippy wool. The major problem for the dyer is the optical one; it must be established that the finer the denier, the more dye required for a given depth of shade.

K. S. Campbell

Text. Research J. May 1947

Dyeing Vinyon

The dyeing of Vinyon. J. A. Woodruff. *Am. Dyestuff Reptr.* **35**, 194–7, 204–5 (Apr. 22, 1946).

The dyes used on Vinyon are divided into classes in accordance with their method of absorption or adsorption on this material. Class I is for dyes which tend to dye Vinyon by solid solution. Class II is for dyes which can be applied by the use of swelling agents for the Vinyon. Class III includes dyes which have an affinity for the surface of Vinyon filaments due to their polar structure. Methods of application for all types of commercial dyes are given. It is stated

that the dyeing of this material has not reached the efficiency achieved in dyeing the natural fibers or rayons. J. A. Woodruff

Text. Research J. May 1947

Wool Dyeing

Dyeing cheeses. Fred Illingworth. *Dyer* **97**, 171–5 (Feb. 14, 1947).

A review of the methods, machines, and dyes used in dyeing woolen and worsted yarns.

Text. Research J. May 1947

Dyeing Colored Wool

Dyeing self-colored wool and hair. Anon. *Dyer* **96**, 543–6 (Dec. 6, 1946).

Practical information is given on the use of chrome and acid milling dyes to cover the color of natural pigments in wool. The problems of obtaining bright shades is emphasized. Dyes are recommended for selected shades and fastness requirements. J. A. Woodruff

Text. Research J. May 1947

Control Instruments in Dyeing

Control instruments and their application to textile dyeing. W. H. Ridley. *Am. Dyestuff Reptr.* **35**, 226–9 (May 6, 1946).

A review.

Text. Research J. May 1947

Printing

Printing cotton fabrics with basic dyestuffs. Anon. *Dyer* **96**, 591–3 (Dec. 20, 1946).

A review.

Text. Research J. May 1947

Printing Mixed Fabrics

The printing of mixed fabrics. G. A. Northup. *Am. Dyestuff Reptr.* **35**, P6–7, P12 (Jan 13, 1947).

The printing of celerandum (viscose-acetate warp, viscose filling), spun viscose-acetate blends, saponified cloth, various other fiber blends, and the discharging of acetate fabrics are discussed. The most important mixed fabric is probably the spun viscose-acetate blend, containing from 25–60% acetate, with warp and filling the same; it is low in price, looks like a wool mixture, and has good stability and hand because of the acetate fiber. Most often the acetate is left white, the fabric being dyed with direct and developed colors chosen to stain the acetate least; it is then printed with vat color patterns. On some lighter qualities paper-back grays, run either once or twice, are used instead of cotton. Much mixed fiber cloth is saponified on the reel and dyed with direct or developed colors before vat printing. All saponified cloth has a tendency to "heat up" if left to stand before ageing, and damage from this source must be guarded against. Air-dried goods have been found to print considerably better than can-dried goods. While vat colors are predominantly used in the printing of mixed fabrics, some spun rayon-cotton mixtures have been printed with the Rapidogens and Indigosols. Most cotton-spun rayon-mohair drapery fabrics are screen-printed with fast-to-light direct or chrome mordant colors. Although Aralac and wool are tendered by the alkali in vat-color pastes they are strong enough when blended with rayon; these blends require careful washing to prevent soaping-off into the ground. Recently, cotton-acetate mixtures (15% acetate to give the appearance of wool when dyed) and very heavy 50–50 cotton-viscose mixtures have been printed. Since acetate was more readily available for dress goods than viscose, during the war, much work in improving discharge printing on acetate was done. At present large quantities of all acetate and acetate mixtures are discharge-printed in single-color white using Decroline.

K. S. Campbell

Text. Research J. May 1947

Printing and Finishing

The influence of advances in other industries on textile printing and finishing. R. J. Hannay. *J. Soc. Dyers and Colourists* **62**, 173–5 (June 1946); *Am. Dyestuff Reptr.* **35**, 352–4 (July 15, 1946).

The influence of work in other industries on the development of new fibers and on the methods of treatment of problems encountered in drying, dyeing, and finishing these fibers is reviewed. J. A. Woodruff

Text. Research J. May 1947

Textile Printing

A quarter of a century of textile printing. P. J. Wood. *Am. Dyestuff Reptr.* **35**, 655–61 (Dec. 2, 1946).

A review.

Text. Research J. May 1947

Reflectance of Dyed Fibers

The cellulose-dyestuff complex. Part III—The effect of orientation on the reflection of polarized light. J. M. Preston and P. C. Tsien. *J. Soc. Dyers and Colourists* **62**, 368–72 (Dec. 1946).

Fiber anisotropy and the resultant dichroism of dyed fibers are shown to affect the relationship between dye concentration and the intensities of reflection of polarized and unpolarized light. Fiber shape and structure are also shown to influence the reflection of light from dyed fibers. Authors

Text. Research J. May 1947

Crocking in Wool

Studies on wool dyeing: Crocking. H. E. Millson, W. H. Watkins, and G. L. Royer. *Am. Dyestuff Reptr.* **36**, P45–63, P69–70 (Feb. 10, 1947).

The microscopical studies discussed in this paper all show that the dye which rubbed off was located on the outer parts of the wool fibers. Dyeings that were made to determine the effect of dye concentration showed that as the concentration of the dye was increased, the crocks became heavier, and an increasingly longer boiling time was required for the available dye to penetrate and gradually reduce the depth of crock. Any excess of dye which cannot penetrate the fibers within a normal dyeing cycle is easily removed during a crock test. Even well-dyed medium shades

produced with acid colors often show some crock. This type of crock cannot be attributed to improper methods of application when the dye is applied by methods which have proved satisfactory over a long period. Electron photomicrographs of the surfaces of dyed wool showed that deposits of dye were on the surface of the fibers before the boil was reached, and concentrated in the areas where the scales joined. Apparently, this dye redissolved with time or increase in temperature, either in the wool fibers directly or in the water phase surrounding the fibers, and penetration was accomplished. However, some crock was obtained in most cases because not all of the dye redissolved and some of it remained on the surface throughout the entire dyeing operation. The greater the number of dye particles remaining, the heavier the crock; this appears to vary with the individual dye and the method of application. The old, established methods for the correction of crocking faults generally employ mechanical action, such as squeeze rolls and fulling mills, or agents such as soaps and alkalies, all of which have a beneficial effect in removing loose dye from materials. Authors

Text. Research J. May 1947

Research Problems in Dyeing and Finishing

Let the Society take up the challenge. C. M. Whittaker. *J. Soc. Dyers and Colourists* **63**, 37–40 (Feb. 1947).

A discussion of problems in the dyeing and finishing industry today which research has created, and problems which research should be encouraged to solve.

K. S. Campbell

Text. Research J. May 1947

Processing Synthetics

Problems connected with dyeing and finishing synthetic fabrics. Weldon Helmus. *Am. Dyestuff Reptr.* **36**, P2–5 (Jan 13, 1947).

Average figures for 1 month's utility requirements for a small dye plant producing 2,000,000 yds. a mo. are given: 15,000,000 gal. of water, 300,000 cu. ft. of gas, 100,000 kw. hrs. of electricity, and 10,000,000 lbs. of steam. Various problems of the synthetic fabric finishes are discussed. One difficulty noted is the variation in dye affinity of yarns, both viscose and acetate, from one manufacturer's product to another. Other problems are due to requirements of the buyer (as to fastness, resin content, etc.) which seem unreasonable in view of actual service requirements. With respect to the importance of proper boil-off, experience has shown that a low-titer soap, used with a small quantity of synthetic detergent and kept at the proper pH with mild alkali, is the best scouring and setting bath for all types of synthetic fabrics. K. S. Campbell

Text. Research J. May 1947

Fabric Finishing

Fabric finishing. R. W. Powell. *Am. Dyestuff Reptr.* **35**, 13–15, 17–18 (Jan. 13, 1946).

A review of modern fabric finishing methods. The continuous peroxide-bleaching process, the du Pont pad steam continuous vat-dyeing process and the Williams unit for the same purpose, resin-emulsion pigment-dyeing, durable water-repellent and other permanent finishes, and fabric stabilization processes are among those described.

K. S. Campbell

Text. Research J. May 1947

Fireproofing Textiles and Papers

The fireproofing of textiles and papers. Norman C. Jones. *Quart. Inst. Fire Engrs.* **6**, 98–113 (1946) (through *Chem. Abstr.* **41**, 1846*f* (Mar. 20, 1947)).

A detailed survey.

Text. Research J. May 1947

Resin Finishing

Twenty-five years of textile resin finishing. J. E. Lynn. *Am. Dyestuff Reptr.* **35**, 653–4 (Dec. 2, 1946).

A review.

Text. Research J. May 1947

Shrinkage Control

Control of shrinkage and crease-resistance of viscose rayon fabrics. H. F. Creegan. *Am. Dyestuff Reptr.* **35**, P514–18 (Nov. 4, 1946).

The causes of rayon fabric shrinkage are discussed. It is suggested that progressive shrinkage on repeated launderings is due to the fact that individual fibers are continuously shifting during wetting and drying cycles to positions of less strain but, because of twist, fabric construction, etc., are unable to release all of the strains in all of the fibers. Three methods of obtaining reasonably permanent, low residual shrinkage—*viz.*, use of resins, the "Definized" process (essentially treatment with 40% NaOH followed by immediate neutralization), and the Cluett Peabody glyoxal process—are described in some detail. In all methods it is essential that maximum relaxation of both filling and warp yarns be allowed in each step up to the actual setting operation.

K. S. Campbell

Text. Research J. May 1947

Unshrinkable Wool

The use of polymers to make wool unshrinkable. Part 2. Di-isocyanates. T. Barr, C. W. Capp, and J. B. Speakman. *J. Soc. Dyers and Colourists* **62**, 338–45 (Nov. 1946).

Di-isocyanates failed to produce unshrinkable wool by cross-linking activity, but did so because of the masking effect on the scale structure of a deposit of polymer resulting from the copolymerization of di-isocyanate and diamine, the diamine being formed by partial conversion of the di-isocyanate in re-

acting with the adsorbed water in the wool. Further development on these lines led to the production of a high degree of unshrinkability in flannel, when 16% by weight of *m*-phenylene di-isocyanate and hexamethylene diamine was applied from solution in benzene, followed by baking 1 hr. at 100° C.

K. S. Campbell

Text. Research J. May 1947

Wool Finishing Machinery

Modern trends in textile machinery. E. Cannity. *Am. Dyestuff Reptr.* **36**, P25–31, P35 (Jan. 27, 1947).

A description of modern wool preparation and wet-finishing machines for woolen and worsted goods. Among those included are the tandem open-width soaper, the continuous fulling mill, the continuous cloth washer, and the carbonizing range. K. S. Campbell

Text. Research J. May 1947

Woolen and Worsted Dyeing and Finishing Machinery

Twenty-five years' progress in woolen and worsted dyeing and finishing machinery. W. von Bergen. *Am. Dyestuff Reptr.* **35**, 662–8 (Dec. 2, 1946).

A review.

Text. Research J. May 1947

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Alginates

The Alginates—I. K. W. L. Kenchington. *Fibres* **8**, 44–8 (Feb. 1947).

A review of the history of alginic acid and of the commercial methods of obtaining it from seaweed. Calcium alginate rayon is produced by spinning an 8% solution of sodium alginate into a bath containing acidified calcium chloride solution, a vegetable oil, and cetyl pyridinium chloride. Alkali-insoluble types are best prepared by treatment of a fabric of calcium alginate with acid and then with basic beryllium acetate or basic chromium alginate. The beryllium salt, being colorless, is most desirable. Data are given on the water vapor absorption, tenacity, and density of a number of the alginate rayons.

Text. Research J. May 1947 A. R. Martin

Cellulose Degradation

The effect of ultraviolet light on cotton cellulose and its influence on subsequent degradation by microorganisms. R. P. Wagner, Harold H. Webber, and Ralph G. H. Siu. *Arch. Biochem.* **12**, 35–50 (Jan. 1947) (through *Bull. Inst. Paper Chem.* **17**, 222 (Jan. 1947)).

The Pressley Index of tensile strength of cotton fibers has been demonstrated to be a useful and convenient measure of the effect of ultraviolet irradiation and fungal action on cotton fibers. As measured by the Pressley Index, the loss of tensile strength of cotton fibers caused by the action of ultraviolet light is independent of oxygen concentration and humidity, but is positively correlated with ultraviolet light intensity and temperature. There is a reduction in fiber tensile strength of approximately 20% after 4 hrs. of exposure to ultraviolet radiation of an intensity of 1,060 mw./sq. cm. Iron and manganese salts catalyze the degrading effect of ultraviolet light on cotton cellulose. The ultraviolet portion of the spectrum, as compared with the visible band, is most effective in modifying the cotton fiber as measured by changes in tensile strength. Exposure of cotton fibers to ultraviolet light increases their resistance to subsequent attack by *Metarrhizium glutinosum*. During the first 4 days of fungal growth, the average rate of degradation of the nonirradiated fibers was twice that of the irradiated fibers. There was no difference in the rate of fungal attack on fibers which were irradiated for the same length of time and with the same light intensity, but with different oxygen concentrations and humidities. 13 references.

Text. Research J. May 1947

Cotton Immaturity and Character

Effect of immaturity on the character of cotton fiber, yarn, and seed. Frank M. Eaton, E. W. Lyle, J. T. Rouse, Geo. W. Pfeiffenberger, and W. H. Tharp. *J. Am. Soc. Agron.* **38**, 1018–33 (1946) (through *Chem. Abstr.* **41**, 1279*h* (Mar. 10, 1947)).

In addition to physical changes due to immaturity, the normal concentration in the lint was increased, the oil concentration in the seed was reduced, and there was a reduction in the weight of lint, oil, and protein per seed. The protein concentration in the seed was unchanged.

Text. Research J. May 1947

Indian Cotton Linters

The analysis, grading, and utilization of Indian linters. L. Thoria and Nazir Ahmad. *Indian Central Cotton Comm. Technol. Lab., Technol. Bull. Ser. B*, No. **34**, 24 pp. (1943) (through *Chem. Abstr.* **41**, 291*b* (Jan. 10, 1947)).

Chemical analyses are given of Indian cotton linters for the 1938–39 and 1939–40 seasons. For the 1938–39 season the ash content of the raw linters was 2.0–11.3% and exceeded 3% in most cases, while only 2 samples of the 1939–40 crop contained more than 3% ash. Linters containing less than 3% ash gave a chemical cotton with an ash content below 0.12%. Linters containing up to 17.5% trash yielded chemical cotton with an α-cellulose content of not less than 98%, the permissible minimum for rayon manufacture. Linters having a trash content of 17.5–32.5% yielded chemical cotton containing 94–98% α-cellulose, while chemical cotton prepared from linters containing 32.5–47.5% trash possessed an α-cellulose content of 85–91%. A relatively low trash content in the form of undeveloped or crushed seed may persist during chemical treatment and may lower the α-cellulose content beyond the industrially permissible limit, whereas a relatively high trash content in the form of leaf, stalk bits, etc., would be re-

moved in the chemical treatment and may not appreciably affect the α-cellulose content of the chemical cotton. Certain linters which were usually high in wax (0.64–0.88%) yielded chemical cotton with a wax content well below the tolerance limit of 0.34%. Study of the relationship between total fiber and α-cellulose showed that the latter can be estimated closely in Indian linters by means of the equation: $\alpha = F + f - K$, where α is the percentage of α-cellulose in air-dry linters, $F + f$ is the percentage of total fiber, and K has the value 16.9.

Text. Research J. May 1947

Drying of Textiles

Some aspects of drying and heating of textiles. Part I—The moisture in fabrics. J. M. Preston and J. C. Chen. *J. Soc. Dyers and Colourists* **62**, 361–4 (Dec. 1946). Part II—The use of infrared radiation, *ibid.*, 364–8 (Dec. 1946).

The "critical moisture content," or condition existing when fibers are just saturated with liquid water without free water on the surfaces, is important because it defines the state of the material at which the drying rate suddenly changes. Before this point is reached the drying rate is constant, and above this point the rate starts to fall. Theoretically, when the critical moisture content is reached there should be a sharp rise in fiber or fabric temperature and experiments showed this to be so; this constitutes therefore a convenient means for determining the critical moisture content. The time at which the temperature rise began was noted, and the percent regain of the material at this time was read from the regain-time curve obtained simultaneously with the use of an automatic recording balance. The expectation that the critical moisture contents would be generally higher than the saturation regains (i.e., the intersection of the absorption isotherm with the 100% R.H. axis) was not confirmed. Critical moisture contents calculated for various fibers were (in g. water per 100 g. dry fibers): wool 39, viscose 38, silk 27, cotton 26, asbestos 7, and glass 6. Measurements and calculations of the absorption of radiation by fabrics by (1) an optical method and (2) from heat transfer measurements, both of which are described, gave substantially similar results. Fabrics absorb considerably less energy when dry than when wet. A given fabric temperature can be attained with a lower flux density with lower temperature (e.g., 600°K) than with higher temperature radiation (e.g., 2000°K); a further advantage of the former is that the presence of dyes makes no difference in the absorption because dyes have negligible absorption in the spectrum region where most of the low temperature radiation is emitted. This may be important since with high temperature sources a dyed pattern could be overdried before the ground was dried. Some large-scale drying experiments showed gas-fired infrared to be less efficient than can-drying or hot-air-drying; it is suggested that infrared must justify itself on the basis of quick starting, flexibility, character of the finish produced, etc.

K. S. Campbell

Text. Research J. May 1947

Latex Treatment of Tire Rayon

Latex treatment of tire rayon. M. M. Heywood. *Trans. Inst. Rubber Ind.* **22**, 159–60 (1946) (through *Chem. Abstr.* **41**, 1865*e* (Mar. 20, 1947)).

A brief review of current processes of particular importance.

Text. Research J. May 1947

Mercerization

Mercerization of cotton fabrics. I. II. Florencia Hernández de Rembado and Alfredo A. Rembado. *Industria y quím.* **8**, 175–85 (1946) (through *Chem. Abstr.* **41**, 1845*f* (Mar. 20, 1947)).

The shrinkage of fabrics on mercerization was a maximum in NaOH solutions of 30° Bé. Increasing time or decreasing temperature of mercerization increased the shrinkage, as did the addition of a humectant (Mercerol B.P.) consisting of phenol, mixed cresols, and a small amount of a high-molecular-weight alkyl sulfate. The absorption of NaOH by the mercerized cotton fibers did not vary in any consistent manner with either the concentration of the NaOH solution or the fiber tension. The absorption of $Ba(OH)_2$, *I*, or the dye Chicago Blue 6B increased to a maximum for fibers mercerized in 30°–33° Bé. NaOH solutions. The absorption of these substances increased to a limiting value with increasing time or temperature of mercerization, or increasing concentration of humectant, and decreased with increasing tension during mercerization. Addition of Na_2CO_3 to the mercerizing bath had no consistent effect on dye absorption. The moisture regain closely paralleled the *I* absorption. Experiments with purified cellulose showed the absorption at equilibrium to be 47.54 milliequivalent NaOH or 69.20 milliequivalent $Ba(OH)_2$ per equivalent cellulose.

Text. Research J. May 1947

Setting of Nylon

The setting of Nylon 66. Vincenzo Navarra. *Materie plastiche* **12**, 69–73 (1946) (through *Chem. Abstr.* **41**, 1445*g* (Mar. 10, 1947)).

Articles made from Nylon 66 are subjected to wet or "dry" heat-treatments to modify their mechanical properties (for bristles and filaments) or to stabilize their shape (e.g., to set the twist of a filament, or the shape of stockings or knitted goods). The setting process requires the presence of heat and a liquid or vapor capable of swelling nylon (even in the "dry" process). Steam is used in most cases, H_2O where a temperature below 100° is necessary. The set can be elastic (i.e., without support or under no more than 5 g. tension) or rigid (i.e., with tensions above 5 g., or rigid support). There is no shrinkage or elongation with 5 g. tension. Elastic set results in shrinkage and loss of orientation, which causes an increase in titer (g./9000 m. fiber) and breaking elongation and a decrease in resistance (ratio of g.

breaking load/titer). Rigid set causes increased orientation (because shrinkage is not possible) and resistance, permanent lengthening, and decreased titer and breaking elongation. Setting treatments at 80°–110° are evaluated by the permanence of a set angle (30°) at which a skein is treated and by determination of the loss in twist in 1 hr. after a filament is treated. It is found that the set is obtained more quickly and more completely with increased temperature; however, hydrolysis limits the temperature of safe operation. The set reaches a maximum for a given temperature; further treatment at this temperature is without effect. To reset an article, the treatment must be more severe (i.e., increased temperature, increased tension, or rigidity of operation). A table shows the conditions used industrially.

Text. Research J. May 1947

Processing Synthetic Fibers

A quarter century of progress in synthetic processing. W. F. Brosnan. *Am. Dyestuff Reptr.* **35**, 646–9, 668 (Dec. 2, 1946).

A review of synthetic fiber processing.

Text. Research J. May 1947

Non-Felting Wool

Recent trends in the production of non-felting wool. Henry Phillips. *Dyer* **96**, 201–3, 251–4, 299–303 (Aug. 30, Sept. 13, Sept. 27, 1946).

A discussion is given of the processes used to date in the production of non-felting wool. The importance of differentiation between shrinkage due to strains remaining in a fabric and the shrinkage due to felting is emphasized in order that the shrinkage problems involved may be understood. Several methods of wet and dry chlorination are discussed. A lower loss in weight of wool during treatment is said to give an advantage to the dry chlorination method over the wet chlorination; the latter is said also to be more difficult to apply uniformly. In addition to the chlorine methods the proteolytic enzyme action, the use of alkylated melamine formaldehyde resins, alkali processes, and others are reviewed. The dyeing properties of chlorinated wool are held to be of interest in obtaining two tones in wool fabrics.

J. A. Woodruff

Text. Research J. May 1947

Water-Repellent Fabrics

Properties of water-repellent fabrics. John W. Rowen and Domenick Gagliardi. *J. Research Natl. Bur. Standards* **38**, 103–17 (Jan. 1947).

A review and an analysis of the theory of water-repellency of textile fabrics have been made. The physicochemical basis underlying the wettability, or water-repellency, of treated fabrics is discussed. A survey of the laboratory test methods for evaluating water-repellency of textile fabrics is presented. A study was made of the water-repellent properties of 11 commercial raincoat and 4 military fabrics. For this study 2 of the more recent tests methods were examined, the drop-penetration and the contact-angle tests. Two other, and older, test methods were also studied, the spray-rating and the hydrostatic-pressure tests. Several exploratory observations were made in an attempt to determine the mechanism by which water-repellent fabrics lose their repellency when exposed to rain.

Text. Research J. May 1947

O.T.S. REPORTS *

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Dyes

Dyestuffs manufacturing processes of I. G. Farbenindustrie. F. O. Robitschek. (FIAT Final Rept. 764.) PB 60946, Aug. 1946; 350 pp.; microfilm, $7.00—photostat, $24.00 (through *Bib. Sci. and Ind. Reports* **4**, 852 (Mar. 7, 1947)).

This report presents indices of the dyes manufactured by I. G. Farbenindustrie, the manufacturing details of which are recorded on microfilm. Several of these microfilms have been received by OTS, and they will be abstracted in subsequent issues of the *Bibliography*; the other reels will be reported as they are received. Each of the products listed is an individual chemical and no blends are included. The first part of the document is an alphabetical register of the German dyes; the second part is a systematic manufacture register of the dyes arranged according to dyeing characteristics and color. The two indices contain for each item the German name, percentage of uncut material, percentage of sales-type material, place of manufacture, reel and frame number of the microfilm where its preparation is described. Whenever possible, reference is made to "Schultze's Farbstoff Tabellen." The systematic division of dyes used is of I. G. origin; it comprises 16 main groups, each group representing certain end use and application characteristics. Each of the primary divisions is divided into the various family groups, and each of the family groups again into the specific type within that family. An index to the systematic register precedes that part. The introduction to the report also contains an evaluation of the German industry, including a table which gives production of dyes by type in the years 1937 and 1943. It is also stated that the Astrazon colors seem to present a new type of dyestuff; the Astrazon colors are water-soluble dyes for acetate silk, having lively shades and good fastness to light and washing. The appendices include a bibliography and a list of the Astrazon samples evacuated to Washington. This copy of the report will not reproduce well. Therefore, copies are being reproduced inexpensively. When these are available, this abstract will appear again in the *Bibliography*.

Text. Research J. May 1947

* Copies of these reports may be obtained from the Office of Technical Services, Department of Commerce, Washington 25, D. C. Orders should be accompanied by check or money order payable to the Treasurer of the United States.

MISCELLANEOUS

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West Indies Cotton

The production and characteristics of the world's cotton crops. Part I. The West Indies. E. Lord. *J. Text. Inst.* **37**, T151–79 (Aug. 1946).

The first part of what promises to be a treatise covers in great detail every important aspect of the cotton industry of the West Indies. Historical, economic, and genetical data are given for the cottons grown on each of the important cotton-producing islands. Physical data include mean length, effective length, percent short fiber, average fiber weight, immaturity count, and standard fiber weight on some 18 varieties grown in the area. Data are also given for the spinning qualities of a number of the varieties. These data are given in terms of the "highest standard count," defined as the highest count that can be spun in a combed yarn with a lea skein product of 2250. These values range from 99 for Montserrat S. I., grown on the island of Nevis, to 183 for St. Vincent Superfine. A. R. Martin

Text. Research J. May 1947

Japanese Cotton Spinning

The Japanese cotton industry. W. T. Thornley. *J. Text. Inst.* **37**, P419–21 (Aug. 1946).

A lecture giving data concerning production capacity, types of cotton used, and yarn counts usually spun. In 1934, there were approximately 8,700,000 spindles and the author states that the production capacity of these was equal to 22,000,000 Lancashire spindles. In February, 1946, 2,150,000 spindles were installed and 1,115,000 were operable. A. R. Martin

Text. Research J. May 1947

Informative Labeling

The informative labeling of textiles. A 25-year review of pros and cons. H. F. Herrmann. *Am. Dyestuff Reptr.* **35**, 616–17, 620 (Dec. 2, 1946).

Text. Research J. May 1947

Lace

The lace industry. W. I. Taylor. *J. Text. Inst.* **37**, P421–9 (Aug. 1946).

A lecture giving a detailed description of the Levers machine.

Text. Research J. May 1947

Textile Chemical Specialties

Textile chemical specialties in the United States from 1921 to 1946. C. Z. Draves. *Am. Dyestuff Reptr.* **35**, 641–4, 661 (Dec. 2, 1946).

A review of the development of textile auxiliaries in the U. S. beginning with the textile uses of soap in 1921. Composition and uses of many proprietary products are described. K. S. Campbell

Text. Research J. May 1947

Textile Research and Education

Trends in textile research and education. W. F. Luther. *Am. Dyestuff Reptr.* **35**, P702–3, P714 (Dec. 30, 1946).

The present status of textile research is reviewed. Institutions engaged in textile education and/or research are listed and briefly described.

Text. Research J. May 1947

PATENT REFERENCES

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Modified Cellulose

Cellulose solution and method of making the same. Frank C. Hewitt and Charles Roy Cayen (to The Aspinook Corp.). U. S. 2,416,998 (Mar. 4, 1947).

Cellulose is degraded with dichromate and dissolved in a solution of sodium zincate in 6*N* NaOH.

Text. Research J. May 1947 A. R. Martin

Coated Fabrics

Manufacture of coated fabric. Paul Rolland Austin (to E. I. du Pont de Nemours & Co., Inc.). U. S. 2,416,041 (Feb. 18, 1947).

Nitrocellulose-coated fabrics are given a top coating of a polyamide. It is claimed that splitting and cracking of the nitrocellulose coating is minimized by this top coat.

Text. Research J. May 1947 A. R. Martin

Non-Woven Fabrics

Composite fabric. Beardsley Lawrence, Jr. (to Utility Fabrics Co., Inc.). U. S. 2,410,884 (Nov. 12, 1946).

Composite fabrics are produced by combining fibrous webs and bonding them together with a thermoplastic binder. A. R. Martin

Text. Research J. May 1947

Conditioning Treatment

Treatment of textile materials. George W. Seymour and Walter Brooks (to Celanese Corporation of America). U. S. 2,406,408 (Aug. 27, 1946).

A yarn or fiber finish is prepared by reacting a mixture of a mineral oil, a vegetable oil, a higher fatty acid, and fuming sulfuric acid at a temperature below 20°C and neutralizing the acid.

Text. Research J. May 1947 A. R. Martin

Crepe Effects

Fabric treatment. Cyril M. Croft and William J. Cramer, Jr. (to Celanese Corporation of America). U. S. 2,410,867 (Nov. 12, 1946).

Crepe effects are produced in cellulose acetate by subjecting the fabric containing crepe-twisted yarns to a 2% acetic acid bath at 90°–99°C for 1 hr. A. R. Martin

Text. Research J. May 1947

Fiber Bonding

Method of manufacturing articles from fibrous material. Bruno Jablonsky. U. S. 2,407,833 (Sept. 17, 1946).

A method of manufacturing shaped articles from fibrous material by

bonding under pressure with an adhesive in a high-frequency electric field. A. R. Martin
Text. Research J. May 1947

Fiber Treatment

Impregnation of regenerated cellulose fibers. George S. Radford and Ira S. Hurd (to Röhm & Haas Co.). U. S. 2,415,564 (Feb. 11, 1947).

Rayon staple is treated with methylol urea solution at 55°F, hydroextracted and dried at 180°F. The monomer within the fiber is then cured by heating for 3 min. at 315°F. An increase in resiliency of 26% is claimed without serious embrittlement of the fibers occurring. A. R. Martin
Text. Research J. May 1947

Flameproofing

Flame- and fireproofing of textile materials. George W. Seymour and George C. Ward (to Celanese Corporation of America). U. S. 2,415,112 (Feb. 4, 1947).

Cellulose acetate is flameproofed by treatment with aqueous solution of 5–25% ammonium bromide and 5–30% urea. Other claims cover the use of diammonium methyl phosphate with the above ingredients. A. R. Martin
Text. Research J. May 1947

Flameproofing textile materials. George W. Seymour and George C. Ward (to Celanese Corporation of America). U. S. 2,415,113 (Feb. 4, 1947).

Cellulose acetate is flameproofed by treatment with 5–20% of a diammonium alkyl phosphate and 2–20% of an ammonium salt, percentages based on weight of textile material. A. R. Martin
Text. Research J. May 1947

Jute Textiles

Treating jute fibers. Roger Wallach (to American Viscose Corp.). U. S. 2,416,272 (Feb. 18, 1947).

The nap fibers of a jute textile are bound to the yarn surface by treating with an alkali-soluble cellulose ether at pH 8, and subsequently coagulating the ether onto the fibers.
Text. Research J. May 1947 A. R. Martin

Removal of Metallic Particles from Card Web

Arrangement for extracting metal bodies from webs of textile materials. Hubert Duesberg (Alien Property Custodian). U. S. 2,416,293 (Feb. 25, 1947).

An electromagnet is placed beneath the web near the doffer comb of a card to remove metallic particles.
Text. Research J. May 1947 A. R. Martin

Continuous Spinning

Process for the manufacture of synthetic yarn. H. O. Naumann (to North American Rayon Corp.). U. S. 2,416,533, 2,416,534, 2,416,535 (Feb. 25, 1947).

A continuous spinning process for the production of wet-spun synthetic fibers. The yarn is wet-spun, after-treated, sized, dried, twisted, and wound on a package suitable for textile use. The claims are concerned chiefly with the arrangement for liquid treating. Separate patents cover the use of the after-treatment method for any thread or yarn. A. R. Martin
Text. Research J. May 1947

Delustering Process

Delustering and weighting textile materials. Robinson Percy Foulds and John Thompson Marsh (to Tootal Broadhurst Lee Co., Ltd.). U. S. 2,416,988 (Mar. 4, 1947).

Textile is delustered by forming particles of urea-formaldehyde within the fibers. A 2-bath treatment with urea followed by formaldehyde containing acid is employed.
A. R. Martin
Text. Research J. May 1947

Knitting Machine

Knitting machine and method of knitting. F. W. Brown. U. S. 2,410,710 (Nov. 5, 1946).

A sweater-knitting machine having a body portion with a tail and side band. A. R. Martin
Text. Research J. May 1947

Bobbin Cap

Yarn twisting apparatus. Otha A. Hitt (to E. I. du Pont de Nemours & Co., Inc.). U. S. 2,406,768 (Sept. 3, 1946).

An improved bobbin cap for use on an uptwister. A. R. Martin
Text. Research J. May 1947

Tow-to-Top Machine

Apparatus for producing synthetic yarn. Albert J. Signoret (to American Safety Razor Corp.). U. S. 2,406,641, 2,406,642 (Aug. 27, 1946).

A separable attachment to a gill box faller bar for cutting filaments into staple while maintaining control of the fibers. Essentially, this converts the gill box into a tow-to-top machine. A. R. Martin
Text. Research J. May 1947

Novelty Yarns

Yarn. Austin N. Oppenheim (Unique Fibers, Inc.). U. S. 2,416,208 (Feb. 18, 1947).

A yarn containing a mixture of synthetic fibers (15–85%), wool (5–60%), and a blend of fur fibers (10–60%). A. R. Martin
Text. Research J. May 1947

Improved Packaging

Packaging and using yarn. Edward J. Abbott (to Abbott Machine Co.). U. S. 2,415,075 (Feb. 4, 1947).

Yarn is wound by means of suction in a tubular container. The container is perforated at the bottom, and air is drawn through the perforations by means of a pump. The incoming yarn is directed to the area of lowest density and thickness by the difference in air permeability of the mass. After the container is filled, a perforated lid is fitted over it. Liquid treatment of the mass of yarn is then possible by circulation through the container.
Text. Research J. May 1947 A. R. Martin